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SUMMARY OF PROCEEDINGS
OF THE
FORTY-NINTH ANNUAL MEETING
BUFFALO, N. Y., JUNE 24–28, 1946

The following Summary records the action taken by the Society at the Annual Meeting on the Recommendations Affecting Standards appearing in the annual reports of the standing committees. In the individual committee reports attention is called through an editorial footnote to any modifications of the recommendations or where the recommendations of the committee were not approved. In all other instances it is to be understood that the recommendations were approved as submitted by the committee. The new and revised standards and tentatives are published in the 1946 Book of A.S.T.M. Standards, Parts I-A and I-B, II, and III-A and III-B. Except as noted, the reports and papers appears in this volume.

This summary of the Forty-ninth Annual Meeting of the American Society for Testing Materials, held at the Hotel Statler, Buffalo, N. Y., June 24–28, 1946, is a record of the transactions of the meeting including the actions taken on the various recommendations submitted by the standing committees. In all, 25 sessions were held.

The registered attendance of the meeting is as follows: Members present or represented, 978; committee members, 405; guests, 452; total, 1835.

The Proceedings are set forth session by session. There were 56 reports and 84 formal papers presented. The record with respect to each has been briefed, the recommendations in the reports having been grouped so as to cover the acceptance of material for publication as tentative, such as new specifications, methods of test, revisions of tentatives and proposed revisions of existing standards, and as a separate group, the approval of matters that were referred to letter ballot of the Society, comprising the adoption of tentatives as standard and the adoption of revisions of standards. Accordingly, wherever the action is reported as "adopted as standard" or "adopted as standard, revisions in" it is understood that this indicates approval of the Annual Meeting for reference to letter ballot of the Society. The various recommendations so recorded are included in the Society letter ballot.[1] The actions designated as "accepted as tentative" or "accepted as tentative, revisions in," are self-evident as indicating acceptance by the Society at the Annual Meeting for publication as tentative.

While all of the items on the program are recorded under the particular session in which they were presented, for convenience in locating actions with respect to any particular report, the accompanying list is presented of all the reports together with the page reference where the action thereon is recorded.

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[1] The letter ballot on recommendations affecting standards, distributed to the Society membership, was canvassed on September 16, 1946 and all actions were approved.

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FIRST SESSION—OPENING SESSION; SYMPOSIUM ON BEARINGS

MONDAY, JUNE 24, 8 P.M.

SESSION CHAIRMAN: PRESIDENT J. R. TOWNSEND

Opening Remarks by President Townsend

President J. R. Townsend extended a very cordial welcome to the members and guests in opening the Forty-ninth Annual Meeting of the Society. He stated that we have one of the most active programs before us at this meeting that the Society has ever planned, pointing out that in all there were eight symposiums to be presented in addition to the regular technical papers and reports. Mr. Townsend also commented on the exhibit that was being held in conjunction with the Annual Meeting which he pointed out was the largest exhibit ever to be held at an Annual Meeting.

Minutes of Forty-eighth Annual Meeting

The Minutes of the Forty-eighth Annual Meeting were approved as published in the 1945 *Proceedings*.[2]

Executive Committee

This report, presented by the Executive Secretary C. L. Warwick, covered a review of membership, publications, finances, new headquarters, and administrative matters dealing with committee activities and the Society's relations with various other organizations. The report points out the policies adopted and put in effect upon the basis of Study Committee recommendations described in the two preceding annual reports. The report also describes in some detail the new administrative organization of the Society which includes the establishment of administrative committees as recommended by the Study Committee. Reports on the progress of work of the present six committees were presented as follows:

- Administrative Committee on Standards (formerly Committee E-10)
- Administrative Committee on Research (formerly Committee E-9)
- Administrative Committee on Papers and Publications (formerly Committee E-6)
- Administrative Committee on Ultimate Consumer Goods
- Administrative Committee on Simulated Service Testing
- Administrative Committee on District Activities

Symposium on Bearings[3]

The Symposium on Bearings consisted of the following five papers:

- Life Testing of Plain Bearings for Automotive Engines—E. T. Johnson, presented by the author.
- Fatigue Testing Machines for Ball and Roller Bearings—Thomas Barish, presented by the author.
- Metallographic Observations of Ball Bearing Fatigue Phenomena—A. B. Jones, presented by the author.
- Fatigue Testing of Roller Bearings—H. R. Gibbons, presented by the author.
- Testing of Bearings Under Controlled Load—J. M. Frankland and C. B. Innes, presented from manuscript by Mr. Frankland.

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[2] Summary of Proceedings of the Forty-eighth Annual Meeting; *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 1 (1945).

[3] Issued as separate publication only.

SECOND AND FOURTH SESSIONS—SYMPOSIUM ON FATIGUE

Second Session

TUESDAY, JUNE 25, 9.30 A.M.

SESSION CHAIRMAN: R. E. PETERSON

- Fatigue Tests of Rail Steel Under Compressive Stress—R. S. Jensen and H. F. Moore, presented by Mr. Moore.
- A Study of the Damaging Effect of Fatigue Stressing on X4130 Steel—J. A. Bennett, presented by the author.

Fatigue of Wood and Glued Wood Constructions—W. C. Lewis, presented from manuscript by the author.

Properties of Several Plastics and an Aluminum Alloy as Obtained from Tests on Four Different Types of Fatigue Machines[3a]—William N. Findley and Robert L. Sutherland, presented from manuscript by Mr. Findley.

Fourth Session

TUESDAY, JUNE 25, 2.00 P.M.

SESSION CHAIRMAN: PAST PRESIDENT H. F. MOORE

Fatigue Tests on Some Additional Copper Alloys—Alton F. Anderson, Emery F. Swan, and Earl W. Palmer, presented from manuscript by Mr. Palmer.

The Fatigue Properties of Beryllium Copper Strip and Their Relation to Other Physical Properties—G. R. Gohn and S. M. Arnold, presented by Mr. Gohn.

The Fatigue Strength of Some Magnesium Sheet Alloys—L. R. Jackson and H. J. Grover, presented by Mr. Jackson.

The Notch Sensitivity in Fatigue Loading of Some Magnesium-Base and Aluminum-Base Alloys—George H. Found, presented from manuscript by the author.

THIRD AND FIFTH SESSIONS—SYMPOSIUM ON SPECTROSCOPIC LIGHT SOURCES[3]

The Symposium on Spectroscopic Light was sponsored by Committee E-2 on Spectrographic Analysis. In addition to the four formal papers, the following men presented discussion on various phases of the subject:

R. H. Bell, Lucius Pitkin, Inc.
Wendell R. Koch, U. S. Army Air Corps.
C. J. Neuhaus, International Nickel Co.
E. K. Jaycox, Bell Telephone Labs., Inc.
J. R. Churchill, Aluminum Company of America.
M. F. Hasler, Applied Research Laboratories.
J. L. Saunderson, Dow Chemical Co.
P. R. Irish, Bethlehem Steel Co.
L. W. Strock, Saratoga Springs Foundation.

Third Session

TUESDAY, JUNE 25, 9.30 A.M.

SESSION CHAIRMAN: E. B. ASHCRAFT

The Present Status of Excitation in Spectrographic Analysis—B. F. Scribner, presented from manuscript by the author.

A Study of the Controlled Spectrographic Spark Source—J. H. Enns and R. A. Wolfe, presented from manuscript by Mr. Enns.

Fifth Session

TUESDAY, JUNE 25, 2.00 P.M.

SESSION CHAIRMAN: H. V. CHURCHILL

Some Properties of Gas Discharges Used as Spectral Sources—R. C. Mason, presented from manuscript by Miss Mary E. Warga.

Short Period Behavior of Spectroscopic Light Sources—G. H. Dieke, presented from manuscript by the author.

FOURTH SESSION—SYMPOSIUM ON FATIGUE

(Continued from Second Session)

TUESDAY, JUNE 25, 2.00 P.M.

This session was a continuation of the Symposium on Fatigue. The papers presented in this session are listed previously with those in the Second Session.

[3a] Publication of this paper temporarily withheld.

FIFTH SESSION—SYMPOSIUM ON SPECTROSCOPIC LIGHT SOURCES

(Continued from Third Session)

TUESDAY, JUNE 25, 2.00 P.M.

This session was a continuation of the Symposium on Spectroscopic Light Sources. The papers presented in this session are listed previously with those in the Second Session.

SIXTH AND ELEVENTH SESSIONS—SYMPOSIUM ON OIL PROCUREMENT PRACTICES[3]

This symposium, sponsored by Technical Committee B of Committee D-2, was held in two sessions, the Sixth and Eleventh Sessions of the Annual Meeting.

Oil procurement practices as used by various industrial concerns vary in complexity from very simple cases, where "viscosity" and "petroleum origin" are the only requirements, to very complex ones incorporating laboratory tests which take a year to complete and field trials as extensive as twelve months before the products under consideration can be evaluated. Such wide variations in procurement practices are to be expected when it is realized that the oils are used to lubricate simple line shafting, in one case and delicate precision machines, in the second.

It is natural for consumers to ask for simple all-inclusive specifications for use in the purchase of lubricants. Such specifications would relieve them of considerable worry attendant to the selection of satisfactory products. Experience has shown that the difficulties encountered in such specifications except in the simplest of cases are not easily surmountable because of the lack of correlation between laboratory test conditions and those encountered in setting up field service. Realizing that complete purchase specifications for oils could not generally be available for many years, the oil and consumer interests cooperatively developed oil procurement practices which have made it possible for the consumer to select the best products for a given job. The practices of several companies were described by the authors participating in the Symposium.

Sixth Session

TUESDAY, JUNE 25, 2.00 P.M.

SESSION CHAIRMAN: W. S. JAMES

SESSION SECRETARY: J. C. GENIESSE

Introduction—T. A. Boyd, presented by the author.

Purchasing of Petroleum Oils by the General Electric Co.—Christian Dantsizen, presented by the author.

Eastman Kodak Purchase Specifications for Lubricants—C. L. Pope, presented from manuscript by the author.

Airline Oil Procurement Practices—J. T. Hendren, presented by the author.

Eleventh Session

WEDNESDAY, JUNE 26, 2.00 P.M.

SESSION CO-CHAIRMEN: T. A. BOYD; W. S. JAMES

SESSION SECRETARY: J. C. GENIESSE

A Recommended Practice for the Purchase of Lubricants—B. C. Voshell, presented from manuscript by the author.

U. S. Navy Oil Procurement Practices—James Costello, presented from manuscript by the author.

Oil Procurement Practice in an Automobile Plant—J. L. McCloud, presented from manuscript by the author.

SEVENTH SESSION—TECHNICAL PAPERS ON METHODS OF TESTING

TUESDAY, JUNE 25, 8.00 P.M.

SESSION CHAIRMAN: P. G. MCVETTY

A Study of the Geometry of the Tension-Impact Specimen—N. A. Kahn and E. A. Imbembo, presented by Mr. Imbembo.

A Study of the Tension Test—Earl R. Parker, Harmer E. Davis, and Alan E. Flanigan, presented from manuscript by Mr. Parker.

A Method for Predicting Failure of Metals[4]—P. E. Cavanagh, presented by the author.

Brinell Hardness of Gray Cast Iron—J. T. MacKenzie, presented from manuscript by the author.

An Automotive Tire Fatigue Machine—J. N. Kenyon, a progress report presented from manuscript by the author on work originally published in A.S.T.M. BULLETIN No. 136, October, 1945, p. 9.

EIGHTH AND TENTH SESSIONS—SYMPOSIUM ON TESTING OF PARTS AND ASSEMBLIES[3]

Sponsored Jointly by A.S.T.M. and the Society for Experimental Stress Analysis

Eighth Session

WEDNESDAY, JUNE 26, 9.30 A.M.

SESSION CHAIRMAN: L. L. WYMAN

The Fatigue Strength of Lap Joints in Some Magnesium Sheet Alloys—H. J. Grover and L. R. Jackson, presented by Mr. Grover.

Fatigue Characteristics of Magnesium Castings—G. H. Found, presented from manuscript by the author.

Endurance—A Criterion of Design—W. T. Bean, presented from manuscript by the author.

Pneumatic Fatigue Machines[4a]—F. B. Quinlan, presented from manuscript by the author.

Tenth Session

WEDNESDAY, JUNE 26, 1.30 P.M.

SESSION CHAIRMAN: CHARLES LIPSON

Automotive Rear Axles and Means of Improving Their Fatigue Resistance—Oscar J. Horger and C. Lipson, presented by H. R. Neifert.

Stress Concentration *versus* the Fatigue Strength of Engine Components—Charles W. Gadd, Andrew Zmuda, and N. A. Ochiltree, presented from manuscript by Mr. Gadd.

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[4] ASTM BULLETIN, No. 143, December, 1946.

[4a] Also published in *Proceedings*, p. 846.

NINTH SESSION—TECHNICAL PAPERS ON CEMENT AND CONCRETE

WEDNESDAY, JUNE 26, 9.30 A.M.

SESSION CHAIRMAN: K. B. WOODS

A Study of Durability and Void Characteristics of Concretes Containing Admixtures, Principally of the Air-Entraining Types—F. B. Hornibrook, Howard Freidburger, and Albert Litvin, presented from manuscript by Mr. Hornibrook.

Permeability of Brick-Mortar Assemblages—John W. McBurney, M. Arnold Copeland, and Russel C. Brink, presented from manuscript by Mr. McBurney.

The Influence of Gypsum on the Hydration and Properties of Portland-Cement Pastes—William Lerch, presented by the author.

Modulus of Elasticity of Aggregates and Its Effect on Concrete—H. A. LaRue, presented from manuscript by the author.

The Use of the Dynamic Modulus of Elasticity in Predicting the 28-Day Flexural Strength of Concrete—E. F. Preece, presented by title only.

A Method of Particle Size Determination of Soils, Cement, etc., by Means of a Chainomatic Specific Gravity Balance—E. V. Barrett, presented by the author.

Concrete Flooring with Asphalt Admixtures[4b]—F. O. Anderegg, presented by the author.

TENTH SESSION—SYMPOSIUM ON TESTING OF PARTS AND ASSEMBLIES

(Continued from Eighth Session)

WEDNESDAY, JUNE 26, 9.30 A.M.

This session was a continuation of the Symposium on Testing of Parts and Assemblies. The papers presented in this session are listed previously with those in the Eighth Session.

Paper:

Investigation of Materials for Marine Propellers[4c]—William C. Stewart and W. Lee Williams, presented from manuscript by Mr. Williams.

ELEVENTH SESSION—PETROLEUM PRODUCTS; SYMPOSIUM ON OIL PROCUREMENT PRACTICES

(Continued from Sixth Session)

WEDNESDAY, JUNE 26, 2.00 P.M.

Committee D-2 on Petroleum Products and Lubricants:

Report presented by T. G. Delbridge, vice-chairman, and the following actions taken:

Accepted as Tentative:

Test for Sulfated Residue from New Lubricating Oils (formerly Emergency Standard ES-43a)

Test for Olefins and Aromatics in Petroleum Distillates (formerly Emergency Standard ES-45a)

Test for Oxidation Stability of Aviation Gasoline (Potential Gum Method)

Accepted as Tentative, Revisions in:

Test for Sulfur in Petroleum Oils by Lamp Method (D 90 - 41 T)

Test for Kinematic Viscosity (D 445 - 42 T)

Test for Sediment in Fuel Oil by Extraction (D 473 - 38 T)

Test for Aniline Point and Mixed Aniline Point of Petroleum Products (D 611 - 44 T)

Test for Knock Characteristics of Aviation Fuels (D 614 - 44 T)

Test for Acid and Base Numbers of Petroleum Products by Color-Indicator Titration (D 663 - 44 T)

Test for Acid and Base Numbers of Petroleum Products by Electrometric Titration (D 664 - 44 T)

Test for Rust-Preventing Characteristics of Steam-Turbine Oil in the Presence of Water (D 665 - 44 T)

Method of Analysis of Petroleum Sulfonates (D 855 - 45 T)

Test for Burning Quality of Kerosine Oils (D 187 - 39)

Adopted as Standard:

Test for Ash Content of Petroleum Oils (D 482 - 43 T)

[4b] ASTM BULLETIN, No. 143, December, 1946.

[4c] Published only in this *Proceedings*, see p. 836.

Test for Oxidation Stability of Gasoline (D 525 - 42 T)

Adopted as Standard, Revisions in:

Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (D 86 - 45)
Test for Flash and Fire Points by Means of Open Cup (D 92 - 45)
Test for Flash Point by Means of the Pensky-Martens Closed Tester (D 93 - 42)
Test for Water in Petroleum Products and Other Bituminous Materials (D 95 - 40)
Test for Water and Sediment in Petroleum Products by Means of Centrifuge (D 96 - 40)
Test for Carbon Residue of Petroleum Products (Conradson Carbon Residue) (D 189 - 41)
Test for Knock Characteristics of Motor Fuels (D 357 - 45)
Test for Gum Content of Gasoline (D 381 - 44)

Editorial Changes Accepted in:

Test for Sulfur in Petroleum Oils by Bomb Method (D 129 - 44), comprising the addition of the following note to Section 1 on Scope: "NOTE.—For the determination of sulfur in additive concentrates or lubricating oils known to contain or suspected of containing additives, Method D 894 - 46 T, Test for Sulfur in Lubricating Oils Containing Additives and in Additive Concentrates by Bomb Method, is to be used."
Test for Acid Heat of Gasoline (D 481 - 39), comprising the addition of the following note to Section 1 on Scope: "NOTE.—A chemical method such as bromine titration or other suitable method is preferred to the acid heat method for determining reactive unsaturation. It is, therefore, recommended that specifications be written in terms of olefin content rather than acid heat. It is anticipated that the acid heat method will eventually be discontinued when it is no longer widely used in specifications."

Accepted for Publication as Information Only:

Test for Sulfated Residue of Lubricating Oils (Indirect Acid-Oxidation Method)
Test for Phosphorus in Organic Materials (Acid-Oxidation Method)
Test for Aromatic Hydrocarbons in Mixtures with Naphthenes and Paraffins by Silica Gel Adsorption

It was announced that the recommendation appearing in the report as preprinted for publication as tentative of the Method of Test for Foaming Characteristics of Viscous Petroleum Oils had been withdrawn by the committee but that, after a minor revision in title and statement of scope, this method would be submitted to the Society through the Standards Committee subsequent to the Annual Meeting.

The committee also withdrew from the report the Proposed Method of Test for Sulfur in Lubricating Oils and in Additive Concentrates by Bomb Method which appeared in the preprinted report as information only. It was announced that this method, after minor revision, would be submitted to the Standards Committee for publication as tentative subsequent to the Annual Meeting.

The recommendation for the immediate adoption of revisions in the Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (D 88 - 44) was withdrawn by the committee from the report.

It was also announced that the committee would present to the Standards Committee after the Annual Meeting a new Tentative Method of Test for Normal Pentane and Benzene Insolubles in Used Lubricating Oils and a new Tentative Method of Test for Oxidation Characteristics of Steam Turbine Oil.

Symposium on Oil Procurement Practices

This was a continuation of the Symposium on Oil Procurement Practices. The papers presented in this session are listed previously with those in the Sixth Session.

TWELFTH SESSION—MARBURG LECTURE; AWARD OF DUDLEY MEDAL

WEDNESDAY, JUNE 26, 4.30 P.M.

SESSION CHAIRMAN: PRESIDENT J. R. TOWNSEND

In introducing the Twentieth Marburg Lecturer, President Townsend recalled that the purpose of the lecture was to have described at annual meetings of the Society by leaders in their respective fields outstanding developments in the promotion of knowledge of engineering materials. Established as a means of emphasizing the importance of one of the functions of the A.S.T.M. in the promotion of knowledge of materials, the lecture honors and perpetuates the memory of Edgar Marburg, first Secretary of the Society, who placed its work on a firm foundation and through his development of the technical programs brought wide recognition to the A.S.T.M. as a forum for the discussion of properties and tests of engineering materials.

President Townsend then presented Mr. J. J. Mattiello, Hilo Varnish Corp., who delivered the lecture on "Protective Organic Coatings as Engineering Materials."[5]

President Townsend, in expressing appreciation to Mr. Mattiello for his instructive and very interesting lecture on a subject of particular interest to the A.S.T.M., presented to him on behalf of the Society, the Edgar Marburg Lecture Certificate.

Award of Charles B. Dudley Medal:

The chairman recognized L. L. Wyman, chairman of the Committee on Award of the Charles B. Dudley Medal. Mr. Wyman presented as the recipient of the Nineteenth Award of the Dudley Medal, Mr. H. R. Copson, Research Chemist, Research Laboratories, The International Nickel Co., Inc., for his paper on "A Theory on the Mechanism of Rusting of Low-Alloy Steels in the Atmosphere" published in the 1945 *Proceedings*. Mr. Wyman stated that this annual award of the Dudley Medal is made to the author of a technical paper of outstanding merit which is considered as constituting an original contribution on research in engineering materials. The Award commemorates the name of the first president of the Society, Charles B. Dudley, and it is also intended as a means of stimulating research in materials and of recognizing meritorious contributions to the Society. On behalf of and as the representative of the Society, President Townsend then made the Nineteenth Award of the Charles B. Dudley Medal to Mr. Copson.

In accepting the medal Mr. Copson expressed sincere appreciation of the honor conferred upon him by this award.

Richard L. Templin Award:

The chairman then announced that the first Richard L. Templin Award has been awarded to R. C. Brumfield, U. S. Naval Test Station, Pasadena, Calif., for his paper on "A Sulfur Print Method for the Study of Crack Growth in the Corrosion-Fatigue of Metals," published in the 1945 *Proceedings*. Due to Mr. Brumfield's inability to attend the Annual Meeting, the award will be at a District Meeting to be held in California sometime in the fall of 1946. The chairman explained that the award was established in 1945 when the Executive Committee received a gift from Richard L. Templin to be used for a prize award for papers describing new testing methods and apparatus. The purpose of the award is to stimulate research in the development of testing methods and apparatus, to encourage the presentation to the Society of papers describing new and useful testing procedures and apparatus, and to recognize meritorious efforts of this character.

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[5] See p. 493.

ANNUAL MEETING DINNER

WEDNESDAY, JUNE 26

TOASTMASTER: THOMAS L. MAYER, ACTING CHAIRMAN, BUFFALO COMMITTEE

The annual meeting dinner, sponsored by the Buffalo General Committee on Arrangements, was attended by 329 members and guests.

At the conclusion of the dinner Mr. Mayer extended a cordial welcome to the Society on behalf of the members in the Western New York—Ontario District. He stated that the District Committee was indeed pleased to serve as host to the Society membership on this occasion of its Forty-ninth Annual Meeting, and expressed the hope that it would prove to be one of the most successful meetings of the Society.

Election of Officers:

The toastmaster than presented President Townsend who recognized W. H. Finkeldey, chairman of the Committee of Tellers, who reported results of the letter ballot on election of officers. Of the 1702 ballots cast, the results were as follows:

For President, to serve for one year:
Arthur W. Carpenter, 1702 votes.
For Vice-President, to serve for two years:
Richard L. Templin, 1702 votes.
For members of the Board of Directors, to serve for three years:
A. G. Ashcroft, 1702 votes.
A. T. Chameroy, 1701 votes.
J. H. Foote, 1699 votes.
F. E. Richart, 1702 votes.
L. H. Winkler, 1702 votes.

The President introduced the newly elected members of the Board of Directors, requesting that they rise as their names were called.

The President then introduced the Vice-President-Elect, Richard L. Templin, who expressed appreciation for the honor conferred on him and for the opportunity given by his election for further service to the Society.

The President then introduced the President-Elect, Arthur W. Carpenter. Mr. Carpenter expressed sincere appreciation of the honor of his election and stated that he realized the responsibilities of the office and that to the best of his ability he would perform the duties of the office to which he had been elected. Mr. Carpenter stated that the coming year may prove to be one of the most important in the history of the Society as a result of the many developments resulting from the war and the need for establishing plans and programs for the future.

Presidential Address:

The toastmaster then presented the President, J. R. Townsend, who delivered the annual presidential address entitled "The Challenge of National and International Affairs to the Engineer."[5a]

Award of Honorary Memberships:

President Townsend then made the award of honorary memberships to the following six members of the Society as they were presented by the Executive Secretary, C. L. Warwick:

W. H. Fulweiler
J. O. Leech (*in absentia*)
J. S. Miller
C. S. Reeve
J. J. Shuman
F. N. Speller

The Executive Secretary reviewed briefly the accomplishments of the recipients and directed particular attention to the contributions or services that they had rendered to the Society. Each of the recipients of honorary membership in turn expressed appreciation of the honor conferred upon him.

Recognition of Forty-Year Members:

The toastmaster recognized the Executive Secretary, C. L. Warwick, who announced that in accordance with action taken by the Executive Committee, certificates were being awarded this year to eight individuals and eleven companies who had attained 40 years of membership in the Society. Presentation of the certificates was then made by the Executive Secretary to the following:

Duff A. Abrams, Consulting Engineer, New York, N. Y.
American Bureau of Shipping, New York, N. Y.
American Steel Foundries, East Chicago, Ind.

[5a] See p. 36.

Anaconda Copper Mining Co., Anaconda, Mont.

Cornell University Library, Ithaca, N. Y.

Oliver C. Cromwell, Former Assistant to Chief of Motive Power and Equipment (Retired), Baltimore & Ohio Railroad Co., Baltimore, Md.

Charles E. Fuller, Professor Emeritus of Theoretical and Applied Mechanics, Massachusetts Institute of Technology, Cambridge, Mass.

Addison F. Holmes, Associate Professor of Applied Mechanics, Massachusetts Institute of Technology, Cambridge, Mass.

The Iron Age, New York, N. Y.

Harry McCormack, Professor of Chemical Engineering, and Director, Department of Chemical Engineering, Illinois Institute of Technology, Chicago, Ill.

Norfolk and Western Railway Co., Roanoke, Va.

Pittsburgh Plate Glass Co., Paint and Varnish Division, Milwaukee, Wis.

Ernest John Russell, Architect, Mauran, Russell, Crowell & Mullgardt, St. Louis, Mo.

Earl B. Smith, Professor of Mechanical Engineering, College of the City of New York, New York, N. Y.

Southern Pacific Co., San Francisco, Calif.

Richard S. Suydam, President, M. B. Suydam Paint Co., Pittsburgh, Pa.

United States Testing Co., Inc., Hoboken, N. J.

University of Kansas Library, Lawrence, Kan.

University of Melbourne Library, Melbourne, Australia

Address by B. K. Sandwell:

The President then presented O. W. Ellis of the Ontario Research Foundation, who introduced the guest speaker, B. K. Sandwell, Editor of *Saturday Night*, Toronto, Ontario, who presented an address entitled "A Testing Time for Canada."

THIRTEENTH AND SEVENTEENTH SESSIONS—ON EFFECT OF TEMPERATURE ON THE PROPERTIES OF METALS

Thirteenth Session—Materials for Gas Turbines[3]

Thursday, June 27, 9.30 a.m.

Session Chairman: N. L. Mochel

Heat-Resisting Metals for Gas Turbine Parts—Howard C. Cross and Ward F. Simmons, presented from manuscript by Mr. Cross.

High-Temperature Alloys Developed for Aircraft Turbosuperchargers and Gas Turbines—J. W. Freeman, E. E. Reynolds, and A. E. White, presented from manuscript by Mr. Freeman.

Chromium-Base Alloys—Robert M. Parke and Frederick P. Bens, presented from manuscript by Mr. Parke.

Metallurgy of High-Temperature Alloys Used on Current Gas Turbine Designs—F. S. Badger, Jr. and W. O. Sweeney, presented from manuscript by Mr. Badger.

Alloys and Ceramic Materials for High-Temperature Service at Battelle Memorial Institute—a talk delivered by Howard C. Cross.

Seventeenth Session—General

Thursday, June 27, 8.00 p.m.

Session Chairman: J. W. Bolton

The Effect of Carbide Spheroidization Upon the Rupture Strength and Elongation of Carbon-Molybdenum Steel—S. H. Weaver, presented by the author.

Resistance of Iron-Nickel-Chromium Alloys to Corrosion in Air at 1600 to 2200 F.—Anton de S. Brasunas, James T. Gow, and Oscar E. Harder, presented from manuscript by Mr. Harder.[6]

Compressive Properties of Aluminum Alloy Sheet at Elevated Temperatures—Alan E. Flanigan, Leslie F. Tedsen, and John E. Dorn, presented by W. A. Reich.[6]

Tensile and Creep Strengths of Some Magnesium-Base Alloys at Elevated Temperature—A. A. Moore and John C. McDonald, presented by Mr. McDonald.[6]

Influence of Strain Rate and Temperature on the Mechanical Properties of Monel Metal and Copper—D. J. McAdam, Jr., G. W. Geil, and D. H. Woodard, presented by L. J. Waldron.

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[6] Also included as part of the Symposium on Materials for Gas Turbines issued as separate publication.

FOURTEENTH AND FIFTEENTH SESSIONS—SYMPOSIUM ON pH MEASUREMENTS[3]

Fourteenth Session

THURSDAY, JUNE 27, 9.30 A.M.

SESSION CHAIRMAN: D. S. MCKINNEY

Historical Review of the Hydrogen Electrode and the Calomel Cell in the Measurement of Hydrogen Ions—C. N. Myers, presented from manuscript by the author.

Definition of pH and Extension of the Acidity Scale to Non-Aqueous Systems—J. C. Warner, D. S. McKinney, and J. P. Fugassi, presented from manuscript by Mr. McKinney.

Standard Buffer Solutions—G. G. Manov, presented from manuscript by the author.

Recent Advances in the Theory and Use of Glass Electrodes for pH Determinations—M. Dole, presented from manuscript by the author.

Fifteenth Session

THURSDAY, JUNE 27, 2.00 P.M.

SESSION CHAIRMAN: D. S. MCKINNEY

Precautions to be Observed in Making Colorimetric pH Determinations—F. R. McCrumb, presented from manuscript by the author.

Acid-Base Equilibria in Aqueous and Nonaqueous Solutions—Martin Kilpatrick, presented from manuscript by the author.

The Potentiometric Estimation of Hydrogen Ion Concentration in Nonaqueous Media—L. Lykken, presented from manuscript by the author.

SIXTEENTH SESSION—FERROUS METALS, PAINTS, AND MISCELLANEOUS SUBJECTS

THURSDAY, JUNE 27, 2.00 P.M.

SESSION CHAIRMAN: L. B. JONES

Committee A-1 on Steel:

Report presented by N. L. Mochel, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Carbon-Steel Plates, Low and Intermediate Tensile Strengths of Structural Quality for Plates 2 in. and Under in Thickness, as a revision and consolidation of Standard Specifications A10 and A78, which latter are to be discontinued.

Spec. for Carbon-Silicon Steel Plates over 2 in. in Thickness of Intermediate Tensile Strength for Machine Parts and General Construction

Spec. for Heat-Treated Alloy-Steel Bars

Spec. for Rolled Carbon-Steel Locomotive Frames

Spec. for Carbon-Chromium Ball- and Roller-Bearing Steels (Formerly Emergency Specifications ES – 5 a).

Accepted as Tentative, Revisions in:

Spec. for Seamless Alloy-Steel Pipe for High-Temperature Service (A 158 – 44 T)

Spec. for Alloy-Steel Bolting Materials for High-Temperature Service (A 193 – 45 T)

Spec. for Seamless Carbon-Molybdenum Alloy-Steel Pipe for High-Temperature Service (A 206 – 44 T)

Spec. for Alloy-Steel Castings Suitable for Fusion Welding for High-Temperature Service (A 217 – 44 T)

Spec. for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 44 T)

Spec. for Mild- to Medium-Strength Carbon-Steel Castings for General Application (A 281 – 45 T), to be redesignated as Tent. Spec. A 27

Spec. for High-Strength Steel Castings for Structural Purposes (A 282 – 45 T), to be redesignated as Tent. Spec. A 148

Spec. for Steel Music Spring Wire (A 228–41)

Adopted as Standard:

Spec. for Carbon-Steel Plates of Flange and Firebox Qualities, Low and Intermediate Tensile Strengths, 2 in. and Under in Thickness,[7] as a revision and consolidation of Standard Specifications A 70 and A 89, which latter are to be discontinued.

Spec. for Heat-Treated Carbon- and Alloy-Steel Track Bolts and Nuts, (A 183 - 40 T), with the following additional change:[7]

Section 4 (*b*).—Change to read as follows: "(*b*) Carbon and manganese determinations shall be made of each melt of bessemer steel, and determinations for phosphorus and sulfur of each melt of bessemer free-cutting and sulfurized nut steel. For other grades of bessemer steel, determinations for phosphorus and sulfur shall be made at intervals not greater than each ten blows, and the next previous determination may be reported."

Adopted as Standard, Revisions in:

Spec. for Open-Hearth Carbon-Steel Rails (A 1 - 39), with the following additional changes:[7]

Section 1.—Change from its present form to read as follows: "1. These specifications cover open-hearth carbon-steel standard Tee rails of nominal weights of 61 lb. per yd. and over. Under special arrangement between the purchaser and the manufacturer, rails of nominal weights of 50 to 60 lb. per yd. may be furnished to the requirements of these specifications for rails of 61 to 69 lb. per yd. except as provided in Section 11(*a*)."

Table I.—Change the present column heading giving weight in pound per yard of 50–69 to read "61–69" with the carbon content, to read "0.55 to 0.68 per cent."

Section 11.—The first line in the Table giving Height of Drop, ft., for the 50 to 60-lb. rails, incl., is to be placed in parentheses with a footnote reading as follows: "This requirement applies under the arrangement referred to in Section 1."

Section 27.—Add a new Section 27 on controlled cooling adopted from the A.R.E.A. Recommended Practice with editorial modifications.

Spec. for Low-Carbon Steel Joint Bars (A 3 - 33), comprising the following changes:[7]

Section 4 (*b*).—Change to read as follows: "(*b*) Carbon and manganese determinations shall be made of each melt of bessemer steel, and determinations for phosphorus and sulfur shall be made at intervals not greater than each ten blows, and the next previous determination may be reported."

Spec. for Steel for Bridges and Buildings (A 7 - 42), with the following additional changes:[7]

Section 1.—Change from its present form to read as follows: "These specifications cover carbon-steel shapes, plates and bars of structural quality for use in the construction of bridges and buildings and for general structural purposes."

Add a note referring to the classification by size of flat-rolled carbon-steel as shown in the Steel Products Manual of the American Iron and Steel Institute with the A.I.S.I. table accompanying the note.

Section 2.—Add the words "sheet, strip" and add a new Paragraph (*d*) to read as follows: "(*d*) Hot-rolled sheets and strip specified to these specifications shall be furnished, unless otherwise specified, to grade C, minimum tensile strength 55000 psi. as prescribed in the Tentative Specifications for Light Gage Structural Quality Flat Hot-Rolled Carbon Steel (0.2499 and 0.1874 in. to 0.0478 in. in Thickness) (A.S.T.M. Designation: A 245)."

Section 9 (*b*).—Change to read as follows. "(*b*) Plates $\frac{3}{16}$ in. in thickness, shapes less than 1 sq. in. in cross-section, and bars, other than flats, less than $\frac{1}{2}$ in. in thickness or diameter need not be subjected to tension tests."

Spec. for Structural Nickel Steel (A 8 - 39), with the following additional changes:[7]

Section 1.—Change to read as follows: "1. These specifications cover high-strength structural nickel steel shapes, plates, and bars up to $1\frac{1}{2}$ in., inclusive, in thickness; intended primarily for special use in main stress-carrying structural members."

Spec. for Wrought Steel Wheels for Electric Railway Service (A 25 - 41),[7] comprising the following changes:

Section 8.—Reletter Paragraphs (*i*) and (*j*) as (*n*) and (*o*) and change to read as follows:

(*n*) *Thickness of Plate.*—The dimensions of the plate may vary but shall not be less than that shown in the Table of Standard Designs, Appendix II.

(*o*) *Diameter of Hub.*—The thickness of wall of finished bored hub shall not be less than that shown in the Table of Standard Designs, Appendix II. The outside diameter shall not exceed that specified by more than $\frac{3}{4}$ in. The thickness of the hub wall in any wheel shall not vary more than $\frac{3}{8}$ in. at any two points eduidistant from the face of the hub.

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[7] These recommendations, not preprinted, were accepted by unanimous consent, subject to favorable letter ballot of Committee A-1, which ballot has been favorable and the results are given in the report of the committee, see p. 104.

Table I.—Change items (*i*) and (*j*) on Thickness of Plate and Diameter of Hub, respectively, in accordance with the above revised Paragraphs (*i*) and (*j*) of Section 8.

Section 9.—Add the following as a new Paragraph (*a*), relettering the remaining paragraphs accordingly: "(*a*) Wheels shall be rough bored and faces of hubs machined. The contour of tread and flange shall be as specified.

Section 10 (*a*).—Change from its present form to read as follows:

10. (*a*) The date (month and year), name or brand of the manufacturer, manufacturer's serial number, and melt number shall be legibly stamped on the back face of the rim approximately $\frac{1}{4}$ in. from the inner edge of the rim. The height of the characters shall not be less than $\frac{5}{8}$ in. for hot stamping nor $\frac{1}{2}$ in. for cold stamping.

Appendices.—Add the following as Appendices I and II:

Appendix I. Spun Steel Wheels: Spun steel wheels are usable for electric railway service similar to wrought steel wheels, providing the spun wheels conform to the foregoing specifications for wrought steel wheels.

Spun steel wheels must be manufactured in a manner capable of producing and maintaining high centrifugal fluid compression forces by a high rate of spinning during the entire period in which solidification of metal takes place.

Appendix II. Wheel Design: The following Tables of Dimensions of Standard Steel Wheel Designs, adopted July 30, 1942, by the American Transit Engineering Association, are hereby made a part of Specifications A 25 – 46. The tables are reprinted with permission. (Insert corrected and expanded Tables I and II from EA – A 25a)

Spec. for Boiler and Firebox Steel for Locomotives (A 30 – 44)

Spec. for Welded and Seamless Steel Pipe (A 53 – 44)

Spec. for Multiple-Wear Wrought Steel Wheels (A 57 – 44), the revised specifications to replace Standard A 57 and Standard A 244.

Spec. for Steel Tie Plates (A 67 – 33), with the following additional changes:[7]

Section 4 (*b*).—Change to read as follows: "(*b*) Carbon and manganese determinations (and copper when specified) shall be made of each melt of bessemer steel, and determinations for phosphorus and sulfur shall be at intervals not greater than each ten blows, and the next previous determination may be reported."

Spec. for Low-Carbon Steel Track Bolts and Nuts (A 76 – 33), comprising the following changes:[7]

Section 4 (*b*).—Change to read as follows: "(*b*) Carbon and manganese determinations shall be made of each melt of bessemer steel, and determinations for phosphorus and sulfur of each melt of bessemer free-cutting and sulfurized nut steel. For other grades of bessemer steel, determinations for phosphorus and sulfur shall be made at intervals not greater than each ten blows, and the next previous determination may be reported."

Spec. for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 – 44)

Spec. for Structural Silicon Steel (A 94 – 39), with the following additional changes:[7]

Section 1.—Change to read as follows: "1. These specifications cover special high-strength structural steel shapes, plates, and bars intended primarily for use in main stress-carrying structural members."

Spec. for Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 105 – 40), comprising the following changes:[7]

Section 4 Manufacturing Practice.—Change Paragraph (*b*) to read as follows: "The material shall be forged by hammering, pressing, rolling, extruding, or upsetting and shall be brought as nearly as practicable to the finished shape and size by hot working and shall be so processed as to cause metal flow during the hot working operation in the direction most favorable for resisting the stresses encountered in service."

Spec. for Commercial Quality Hot-Rolled Bar Steels (A 107 – 42)

Spec. for Commercial Cold-Finished Bar Steels and Cold-Finished Shafting (A 108 – 36)

Spec. for Structural Steel for Locomotives and Cars (A 113 – 42), with the following additional changes:[7]

Section 1.—Change to read as follows: "1. These specifications cover carbon-steel shapes, plates, (except boiler and firebox plates) and bars intended primarily for use in locomotives and car construction."

Section 7 (*b*).—Change to read as follows: "(*b*) Plates $\frac{3}{16}$ in. in thickness, shapes less than 1 sq. in. in cross-section, and bars other than flats, less than $\frac{1}{2}$ in. in thickness or diameter, need not be subjected to tension tests."

Spec. for Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 – 44)

Spec. for Open-Hearth Iron Plates of Flange Quality (A 129 – 39)

Spec. for Structural Steel for Ships (A 131 – 39)

Spec. for Electric-Resistance-Welded Steel Pipe (A 135 – 44)

Spec. for Electric-Fusion-Welded Steel Pipe (Sizes 8 in. to but not Including 30 in.) (A 139 – 42)

Spec. for Seamless Low-Carbon and Carbon-Molybdenum Steel Still Tubes for Refinery Service (A 161 – 44)

Spec. for Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 44)

Spec. for Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 – 44)

Spec. for Forged or Rolled Steel Pipe Flanges for General Service (A 181 – 42),

Section 4 (b).—Change to read the same as recommended above for Section 4 *(b)* of Specifications A 105.

Spec. for Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 182 – 44),

Section 4 (b).—Change to read the same as recommended above for Section 4 *(b)* of Specifications A 105.

Spec. for One-Wear and Two-Wear Wrought Steel Wheels (A 186 – 39), comprising the following changes:[7]

Title.—Change to read "Standard Specifications for One-Wear Wrought Steel Wheels."

Section 1.—Change to read as follows: "1. These specifications cover Class 1 and 2 one-wear wrought steel wheels for use under cars in freight and industrial service."

Section 9g.—Change the second sentence by deleting the phrase: "for one-wear wheels or 2 in. for two-wear wheels."

Make the necessary editorial changes in other sections deleting all reference to two-wear wheels.

Section 10.—Insert a new Paragraph *(b)* to read as follows, relettering the present Paragraphs *(b)* and *(c)* as *(c)* and *(d)*:

(b) Class 1. Rolled to Finish Wheels.—In Class 1 wheels the contour of tread and flange of the wheels may be as rolled or machined at the option of the manufacturer.

Class 2. Machine-Finished Wheels.—In Class 2 wheels the contour of the tread and flange of all wheels furnished must be machine finished.

Spec. for Carbon- and Alloy-Steel Nuts for Bolts for High-Pressure and High-Temperature Service (A 194 – 40)

Spec. for Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 – 44)

Spec. for Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 – 44)

Spec. for Carbon-Silicon Steel Plates of Ordinary Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels (A 201 – 44)

Spec. for Chrome-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels (A 202 – 44)

Spec. for Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 – 44), this item having been recommended in the preprinted report for publication as tentative.

Spec. for Molybdenum-Steel Plates for Boilers and Other Pressure Vessels (A 204 – 44)

Spec. for Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 209 – 44)

Spec. for Medium-Carbon Seamless Steel Boiler and Superheater Tubes (A 210 – 44)

Spec. for High Tensile Strength Carbon-Silicon Steel Plates for Boilers and Other Pressure Vessels (Plates 4½ in. and Under in Thickness) (A 212 – 44)

Spec. for Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 – 44)

Spec. for Manganese-Vanadium Steel Plates for Boilers and Other Pressure Vessels (A 225 – 44)

Spec. for Carbon-Steel Forgings for General Industrial Use (A 235 – 42)

Spec. for Carbon-Steel Forgings for Locomotives and Cars (A 236 – 42)

Spec. for Alloy-Steel Forgings for General Industrial Use (A 237 – 42)

Spec. for Alloy-Steel Forgings for Locomotives and Cars (A 238 – 42)

Spec. for Low-Alloy Structural Steel (A 242 – 42)

Spec. for Carbon-Steel and Alloy-Steel Ring and Disk Forgings (A 243 – 43)

Spec. for Atomic-Hydrogen-Arc-Welded and Electric-Resistance-Welded Alloy-Steel Boiler and Superheater Tubes (A 249 – 44)

Spec. for Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 – 44)

Spec. for Welded Alloyed Open-Hearth Iron Pipe (A 253 – 44)

Spec. for Copper Brazed Steel Tubing (A 254 – 44)

Spec. for Welded and Seamless Steel Pipe Piles (A 252 – 44), comprising the following changes:[7]

Section 1.—Change from its present form to read as follows:

1. These specifications cover furnace-welded, electric-welded, and seamless steel black pipe piles of cylindrical shape. They apply to piles in which the steel cylinder acts as a permanent load-carrying member, or alternatively, acts as a shell to form cast-in-place concrete piles. These specifications

are not intended to apply to pipe for general structural purposes.

Section 4 (a).—Add a new second sentence to read as follows: "By agreement between the manufacturer and the purchaser, the tension test specimen may be taken from the skelp."

New Table.—Add a new Table I to read as shown in the accompanying Table II.

Designating Wall Thickness in Tubing Specifications—to be Minimum Wall Thickness.—One standard method of designating wall thickness of various kinds of tubing has been agreed upon by the interest concerned, and that standard is to be the minimum wall thickness. At present the various specifications provide for the use of either minimum or average wall. In the specifications listed below changes will be incorporated to achieve this end. In general, the revisions comprise a deletion of the reference to average wall thickness. Explanatory notes at the end of some of the specifications will also be modified accordingly. The specifications affected are as follows: A 161 - 44, A 200 - 44, A 271 - 44 T, A 179 - 44, A 199 - 44, and A 214 - 44.

Clarification of Thickness in Ordering Pipe.—In the interest of clarification and to indicate that pipe ordered under the ten specifications listed below is nominal (average) wall thickness a number of editorial changes will be made. Also, added to each specification will be a complete table giving minimum wall thickness on inspection for nominal (average) pipe wall thickness. While a complete table (the accompanying Table I) will be added to each specification, an appropriate note will indicate that this does not imply all of the wall thickness listed can be obtained under any one of the specifications. The designations of the specifications affected are as follows: A 53 - 44, A 106 - 45 T, A 120 - 44, A 135 - 44, A 139 - 42, A 158 - 44 T, A 206 - 44 T, A 252 - 44, A 253 - 44, and A 280 - 46 T.

TABLE I.—TABLE OF MINIMUM WALL THICKNESSES ON INSPECTION FOR NOMINAL (AVERAGE) PIPE WALL THICKNESSSES.

NOTE 1.—The following formula, upon which this table is based, may be applied to calculate minimum wall thickness from nominal (average) wall thickness:

$$t_n \times 0.875 = t_m$$

where:

t_n = nominal (average) wall thickness in inches, and

t_m = minimum wall thickness in inches.

The wall thickness is expressed to three decimal places, the fourth decimal place being carried forward or dropped, in accordance with the Tentative Recommended Practices for Designation of Numerical Requirements in Standards (A.S.T.M. Designation: E 29).

NOTE 2.—This table is a master table covering wall thicknesses available in the purchase of different classifications of pipe, but it is not meant to imply that all of the walls listed therein are obtainable under this specification.

Nominal (Average) Thickness (t_n), in.	Minimum Thickness on Inspection (t_m), in.	Nominal (Average) Thickness (t_n), in.	Minimum Thickness on Inspection (t_m), in.	Nominal (Average) Thickness (t_n), in.	Minimum Thickness on Inspection (t_m), in.
0.068	0.060	0.279	0.244	0.600	**0.525**
0.088	0.077	0.280	0.245	0.625	**0.547**
0.091	0.080	0.281	0.246	0.656	**0.574**
0.095	0.083	0.294	0.257	0.674	**0.590**
0.113	0.099	0.300	0.262	0.687	**0.601**
0.119	0.104	0.307	0.269	0.718	**0.628**
0.125	0.109	0.308	0.270	0.750	**0.656**
0.126	0.110	0.312	0.273	0.812	**0.710**
0.133	0.116	0.318	0.278	0.843	**0.738**
0.140	0.122	0.322	0.282	0.864	**0.756**
0.145	0.127	0.330	0.289	0.875	**0.766**
0.147	0.129	0.337	0.295	0.906	**0.793**
0.154	0.135	0.343	0.300	0.937	**0.820**
0.156	0.136	0.344	0.301	1.000	**0.875**
0.179	0.157	0.358	0.313	1.031	**0.902**
0.187	0.164	0.365	0.319	1.062	**0.929**
0.188	0.164	0.375	0.328	1.125	**0.984**
0.191	0.167	0.382	0.334	1.156	**1.012**
0.200	0.175	0.400	0.350	1.218	**1.066**
0.203	0.178	0.406	0.355	1.250	**1.094**
0.216	0.189	0.432	0.378	1.312	**1.148**
0.218	0.191	0.436	0.382	1.343	**1.175**
0.219	0.192	0.437	0.382	1.406	**1.230**
0.226	0.198	0.438	0.383	1.437	**1.257**
0.237	0.207	0.500	0.438	1.500	**1.312**
0.250	0.219	0.531	0.465	1.562	**1.367**
0.258	0.226	0.552	0.483	1.750	**1.531**
0.276	0.242	0.562	0.492		
0.277	0.242	0.593	0.519		

TABLE II.—TENSILE REQUIREMENTS.

	Grade 1	Grade 2	Grade 3
Tensile strength, min., psi	50 000	60 000	75 000
Yield point, min., psi	30 000	35 000	45 000
Basic minimum elongation for walls 5/16 in. and over in thickness:			
Elongation in 8 in., min., per cent	18	14	....
Elongation in 2 in., min., per cent	30	25	20
For walls less than 5/16 in. in thickness a deduction for each 1/32-in. decrease in wall thickness below 5/16 in. from the basic minimum elongation of the following percentage	1.50[a]	1.25[a]	1.0[a]

[a] The following table gives the computed minimum values:

Wall Thickness, in.	Elongation in 2 in., min., per cent		
	Grade 1	Grade 2	Grade 3
5/16 (0.312)	30.00	25.00	20.00
9/32 (0.281)	28.50	23.75	19.00
1/4 (0.250)	27.00	22.50	18.00
7/32 (0.219)	25.50	21.25	17.00
3/16 (0.188)	24.00	20.00	16.00

NOTE.—The above table gives the calculated minimum elongation values for each 1/32-in. decrease in wall thickness Where the wall thickness lies between two values shown above, the minimum elongation value shall be determined by the following formula:

Grade	Formula
1	$E = 56\,t + 17.50$
2	$E = 40\,t + 12.50$
3	$E = 32\,t + 10.00$

where:
E = elongation in 2 in. in per cent, and
t = actual thickness of specimen, in inches.

Editorial Changes Accepted in:

Spec. for Electric-Fusion-Welded Steel Pipe for High-Temperature and High-Pressure Service (A 155 – 42)

Spec. for Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 – 44 T)

Spec. for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 – 44 T)

Spec. for Seamless and Welded Austenitic Stainless Steel Tubing for the Dairy and Food Industry (A 270 – 44 T)

Withdrawal of Standards:

Spec. for Mild Steel Plates (A 10 – 39)

Spec. for Carbon-Steel Castings for Miscellaneous Industrial Uses (A 27 – 44)

Spec. for Carbon-Steel Plates for Stationary Boilers and Other Pressure Vessels (A 70 – 44)

Spec. for Low Tensile Strength Carbon-Steel Plates of Structural Quality for Welding (A 78 – 43)

Spec. for Carbon-Steel and Alloy-Steel Castings for Railroads (A 87 – 44)

Spec. for Low Tensile Strength Carbon-Steel Plates of Flange and Firebox Qualities (A 89 – 43)

Spec. for Alloy-Steel Castings for Structural Purposes (A 148 – 44)

Spec. for Carbon-Steel Castings Suitable for Fusion Welding for Miscellaneous Industrial Uses (A 215 – 44)

Spec. for Heat-Treated Wrought Steel Wheels (A 244 – 44)

Reaffirmation of Standards:

Spec. for Open-Hearth Steel Girder Rails of Plain, Grooved and Guard Types (A 2 – 27)

Spec. for Medium-Carbon Steel Joint Bars (A 4 – 14)

Spec. for High-Carbon Steel Joint Bars (A 5 – 37)

Spec. for Billet-Steel Bars for Concrete Reinforcement (A 15 – 39)

Spec. for Rail-Steel Bars for Concrete Reinforcement (A 16 – 35)

Spec. for Quenched Carbon-Steel Joint Bars (A 49 – 39)

Spec. for Soft Steel Track Spikes (A 65 – 33)

Spec. for Steel Screw Spikes (A 66 – 33)

Spec. for Cold-Drawn Steel Wire for Concrete Reinforcement (A 82 – 34)

Spec. for Cold-Rolled Strip Steel (A 109 – 38)

Spec. for Axle-Steel Bars for Concrete Reinforcement (A 160 – 39)

Spec. for Fabricated Steel Bar or Rod Mats for Concrete Reinforcement (A 184 – 37)

Spec. for Welded Steel Wire Fabric for Concrete Reinforcement (A 185 – 37)

Spec. for Hot-Worked High-Carbon Steel Tie Plates (A 241 – 41)

Committee A-3 on Cast Iron:

Report presented by J. T. MacKenzie, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Def. of Terms Relating to Cast Iron (A 196 – 42 T)

Adopted as Standard:

Method of Compression Testing of Cast Iron (A 256 – 42 T)

Adopted as Standard, Revisions in:

Spec. for Gray Iron Castings (A 48 – 41)

Withdrawal of Standard:

Spec. for Automotive Gray Iron Castings (A 159 - 41)

Committee A-5 on Corrosion of Iron and Steel:

Report presented by C. D. Hocker, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Lead Coating (Hot-Dip) on Iron or Steel Hardware (A 267 - 44 T)

Adopted as Standard:

Spec. for Zinc-Coated (Galvanized) Iron or Steel Sheets (A 93 - 38 T)

Adopted as Standard, Revisions in:

Rec. Practice for Safeguarding Against Embrittlement of Hot-Galvanized Structural Steel Products and Procedure for Detecting Embrittlement (A 143 - 35).

Withdrawal of Standard:

Spec. for Zinc-Coated (Galvanized) Iron or Steel Sheets (A 93 - 27)

Editorial Change in Standard:

Spec. for Zinc (Hot-Galvanized) Coatings on Structural Steel Shapes, Plates and Bars, and Their Products (A 123 - 33), comprising the addition of the following footnote to Section 5 (*a*): "See Recommended Practice for Safeguarding Against Embrittlement of Hot-Galvanized Structural Steel Products and Procedure for Detecting Embrittlement (A.S.T.M. Designation: A 143)."

It was announced that the Tentative Specifications for Electrodeposited Coatings of Cadmium on Steel (A 165 - 40 T) had been transferred to the jurisdiction of Committee B-8 on Electrodeposited Metallic Coatings. The Tentative Specifications for Electrodeposited Coatings of Zinc on Steel (A 164 - 40 T), referred to in the report as preprinted, are being retained under the jurisdiction of Committee A-5.

Committee A-6 on Magnetic Properties:

Report presented by Thomas Spooner, chairman, and the following actions taken:

Accepted as Tentative:

Test for Core Loss at Frequencies Higher than Power Frequencies and Ductility of Magnetic Materials (A 34 - 46 T)

Adopted as Standard:

Def. of Terms, with Symbols, Relating to Magnetic Testing (A 127 - 44 T), as revised.

Adopted as Standard, Revisions in:

Tests for Magnetic Properties of Iron and Steel (A 34 - 44), with minor editorial changes in the method as preprinted.

Committee A-7 on Malleable-Iron Castings:

Report, not preprinted, presented from manuscript by C. O. Burgess, chairman.

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys:

Report presented by Jerome Strauss, chairman, and the following actions taken:

Adopted as Standard, Revisions in:

Rec. Practice for Conducting Plant Corrosion Tests (A 224 - 41)

Editorial Change Accepted in:

Spec. for Corrosion-Resisting Chromium and Chromium-Nickel Steel Plate, Sheet, and Strip for Fusion-Welded Unfired Pressure Vessels (A 240 - 44)

Report of Committee D-1 on Paint, Varnish, Lacquer, and Related Products:

Report presented by W. T. Pearce, chairman, and the following actions taken:

Accepted as Tentative:

Method of Evaluating Degree of Resistance of Traffic Paint to Bleeding
Test for Evaluating Degree of Settling of Traffic Paint
Spec. for Pumice Pigment
Test for Cellulose Acetate
Test for Changes in Protective Properties of Coatings of Paint, Varnish, Lacquer, and Related Products on Steel Surfaces When Subjected to Immersion (formerly Emergency Standard ES - 35, with editorial changes)
Spec. for Titanium Dioxide Pigments (D 476 - 46 T)

Accepted as Tentative, Revisions in:

Test for Dry to No-Pick-Up Time of Traffic Paint (D 711 - 43 T)
Spec. for Red and Brown Iron Oxide Pigments (D 84 - 43 T)
Spec. for Yellow Iron Oxide, Hydrated (D 768 - 44 T)

Methods of Chemical Analysis of Zinc Yellow Pigment (Zinc Chromate Yellow) (D 444 - 39)

Sampling and Testing Lacquer Solvents and Diluents (D 268 - 44)

Spec. and Tests for Soluble Nitrocellulose (D 301 - 33), with an additional revision providing in Section 11 for the omission of the reference to "No. 5" in the requirements for 188-190 proof denatured alcohol.

Adopted as Standard:

Spec. for Oiticica Oil (Permanently Liquid) (D 601 - 41 T), as revised

Test for Light Sensitivity of Traffic Paint (D 712 - 43 T)

Method of Conducting Road Tests on Traffic Paint (D 713 - 43 T)

Def. of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 - 44 T), as revised

Spec. for Methyl Ethyl Ketone (D 740 - 43 T)

Spec. for Isopropyl Alcohol (D 770 - 44 T)

Test for Water in Lacquer Solvents and Diluents (D 268 - 44 T)

Adopted as Standard, Revisions in:

Spec. for Tricresyl Phosphate (D 363 - 36)

Spec. for Lampblack (D 209 - 41)

Spec. for Bone Black (D 210 - 41)

Spec. for Carbon Black (D 561 - 41)

Spec. for Prussian Blue (D 261 - 41)

Spec. for Ultramarine Blue (D 262 - 41)

Spec. for Chrome Green (D 212 - 41)

Spec. for Chrome Oxide Green (D 263 - 41)

Report of Committee E-1 on Methods of Testing:

Report presented by W. H. Fulweiler, chairman, and the following actions taken:

Adopted as Standard, Revisions in:

Spec. for A.S.T.M. Thermometers (E 1 - 44), except that the committee withdrew from the report the recommendation for the omission from the specifications of the six Saybolt viscosity thermometers graduated in Centigrade, and presented certain other minor changes.

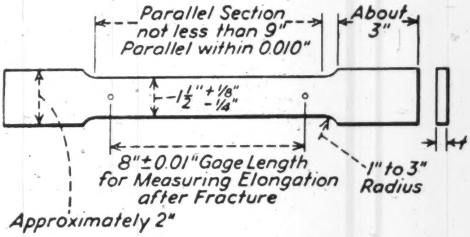
Methods of Tension Testing of Metallic Materials (E 8 - 42),[8] with the following additional modifications (except the revision in Section 19) resulting from recommendations received from Committee A-1 on Steel:

Section 1.—Change the first sentence to read as follows by the addition of the italicized words: "These methods *and the figures which are a part of the methods* cover the apparatus, specimens, and procedures for the tension testing of metallic materials."

New Section.—Add the following new Section 4, renumbering the present sections accordingly:

4. *Test Specimens.*—Improperly prepared test specimens often cause unsatisfactory test results. It is important, therefore, that care be exercised in the preparation of specimens, particularly in the machining, to assure good workmanship.

Fig. 1.—Change the illustration of the 8-in. gage length rectangular tension test specimen to read as follows:


Parallel Section not less than 9"
Parallel within 0.010"
About 3"
$1\frac{1}{2}$" +1/8" -1/4"
t
8" ± 0.01" Gage Length for Measuring Elongation after Fracture
1" to 3" Radius
Approximately 2"

NOTE 1.—When necessary, it is permissible to use a narrower specimen, but in such a case the reduced portion shall be not less than 1 in. in width.

NOTE 2.—Punch marks for measuring elongation after fracture shall be made on the flat or on the edge of the specimen and within the parallel section; either a set of nine punch marks 1 in. apart, or one or more sets of 8-in. punch marks may be used.

NOTE 3.—The dimension "t" is the thickness of the test specimen as provided for in the applicable material specifications.

Section 18.—Renumber as Section 19 and change to read as follows by the addition of the italicized words and figures:

19. *For the test specimen illustrated in Fig. 1, the width should be measured at each end of the gage length to determine parallelism, and also at the center. The thickness should be measured at the center only and used with the center measurement of the width to determine the cross-section area. The center width dimension should be recorded to the nearest 0.005 in., and the thickness measurement to the nearest 0.001 in.* The dimensions of cross-section of *all other* tension test specimens shall be measured by means of a measuring instrument, preferably reading to 0.5 per cent of the dimensions measured and in any case to at least 0.001 in. The stress shall be calculated on the basis of the minimum cross-section as determined by measurement.

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[8] This recommendation, not preprinted, was accepted by unanimous consent, subject to favorable letter ballot of Committee E-1, which ballot has been favorable and the results are given in the report of the committee, see p. 470.

Section 19.—Renumber as Section 20 and make the following changes in this section on Speed of Testing from the form as preprinted: (1) Change the last sentence of Paragraph (*a*) to read "The average crosshead speed can be experimentally determined by using a suitable scale and stop watch." (2) Change the second sentence of Paragraph (*b*) to read "The average rate of stressing can be determined with a stop watch by observing the time required to apply a known increment of stress." (3) In the second sentence of Paragraph (*c*) change "rate of straining" to read "average rate of straining." (4) In the last paragraph add the following as a new second sentence: "The speed above this point shall be within the limits specified." (5) Add the following as a note at the end of the section: "In writing new standards, or in revising old standards, the A.S.T.M. product specifications committee will have the responsibility of deciding for any given material what method of measuring speed of testing is to be used, and of specifying suitable numerical limits for crosshead speed, rate of stressing, or rate of straining."

The proposed revision in Tentative Definitions with Procedures Relating to Conditioning and Weathering (E 41 - 42 T) which appeared in the preprinted report was withdrawn by the committee.

The chairman then called upon H. F. Moore to describe the activities of Technical Committee I on Mechanical Testing, and on Robert Burns to report on the work of Technical Committee X on Conditioning and Weathering.

Committee E-3 on Chemical Analysis of Metals:

Report presented by J. W. Stillman, secretary. It was announced that the large number of new tentative methods and the revised methods accepted earlier in the year through the Standards Committee would be published in the 1946 Book of A.S.T.M. Methods of Chemical Analysis of Metals to be issued in August.

Committee E-4 on Metallography:

Report presented by L. L. Wyman, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Rec. Practice for Determining the Inclusion Content of Steel (E 45 - 42 T), except that the change in Section 11 (*b*) should be changed from the form as preprinted to read as follows:

Section 11 (b).—At the end of the last paragraph add the following as a new paragraph: "If desired, the predominant type of inclusions may be recorded, whether they are sulfides, silicates, or oxides."

Methods of Preparation of Metallographic Specimens (E 3 - 44 T),[8a] with a number of minor changes to bring the methods up-to-date and in accord with best modern practice.

Rec. Practice for Identification of Crystalline Materials by the Hanawalt X-Ray Diffraction Method (E 43 - 42 T),[8b] comprising the addition of the following paragraph to Section 8:

(*b*) *Calculating Interplanar Spacings.*—The measurements described in Paragraph (*a*) are not used directly in the index for the identification of crystalline materials, because the distances between corresponding lines depend upon the radius of the camera and the type of radiation used as well as upon the crystal structure of the sample. The patterns are described uniquely in terms of the interplanar spacings $\left(\frac{d}{n}\right)$ corresponding to the diffraction lines.[a] These interplanar spacings are calculated from the measured positions of the lines by using Bragg's equation:

$$\frac{d}{n} = \frac{\lambda}{2 \sin \theta}$$

where:

$\frac{d}{n}$ = the first order spacing,

λ = wave length of the X-rays, and

θ = diffraction angle. For the cylindrical camera recommended above, θ (in degrees) is calculated using the following equation: $\theta = \frac{45\,l}{\pi r}$ where l is the distance between corresponding lines on opposite sides of the undeviated beam, and r is the radius of the film. Many cameras are made with a radius of 57.3 mm. so that 1 mm. on the film equals 1 degree of arc. The best way to measure r is to make a pattern of a material with an accurately known structure (for example, NaCl) and to calculate *r* from some of the lines of that pattern. When a laboratory makes all powder patterns with the same type of camera and the same type of X-rays, it is well worth the effort to make a scale which reads the interplanar spacings directly from the film.

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[a] All data which are submitted to the committee for inclusion in the index should be in terms of $\frac{d}{n}$.

[8a] This recommendation, not preprinted, was accepted by unanimous consent, subject to favorable letter ballot of Committee E-4, which ballot has resulted in a vote of 30 affirmative, 0 negative, and 7 ballots marked "not voting" from a committee membership of 73.

[8b] This recommendation, not preprinted, was accepted by unanimous consent, subject to favorable letter ballot of Committee E-4, which ballot has resulted in a vote of 29 affirmative, 0 negative, and 9 ballots marked "not voting" from a committee membership of 73.

Adopted as Standard:

Classification of Austenite Grain Size in Steels (E 19 - 39 T)

Committee E-6 on Methods of Testing Building Construction:

Report presented by L. J. Markwardt, chairman.

Joint Committee on Filler Metal:

A progress report, not preprinted, presented by title and accepted for publication in the ASTM BULLETIN, August, 1946.

Committee E-2 on Spectrographic Analysis:

Report, not preprinted, presented from manuscript by Mary E. Warga, secretary.

SEVENTEENTH SESSION—ON EFFECT OF TEMPERATURE ON THE PROPERTIES OF METALS

(Continued from Thirteenth Session)

THURSDAY, JUNE 27, 8.00 P.M.

SESSION CHAIRMAN: J. W. BOLTON

Report of Joint Research Committee on Effect of Temperature on Properties of Metals:

Report presented from manuscript by N. L. Mochel, chairman.

Mr. Mochel then called upon J. J. Kanter to present from manuscript the report on Stability of Steels as Affected by Temperature, which is to appear as an Appendix to the report of the joint committee.

Session on Effect of Temperature on the Properties of Metals

This session was a continuation of the Session on Effect of Temperature on the Properties of Metals. The papers presented in this session are listed previously with those in the Thirteenth Session.

EIGHTEENTH SESSION—SYMPOSIUM ON ATMOSPHERIC WEATHERING OF CORROSION-RESISTANT STEELS

THURSDAY, JUNE 27, 8.00 P.M.

SESSION CHAIRMAN: F. L. LAQUE

Results of 15-yr. Exposure Tests on Corrosion-Resistant Steels—I. V. Williams and K. G. Compton, presented by Mr. Williams.

Atmospheric Corrosion Tests on Corrosion-Resistant Steel—Grant L. Snair, Jr., presented from manuscript by the author.

Atmospheric Corrosion Tests on High-Chromium Steels—W. O. Binder and C. M. Brown, presented by Mr. Binder.

Weathering Behavior of Corrosion-Resistant Steel Insect Screens—W. A. Wesley and H. R Copson, presented by Mr. Copson.

Corrosion-Resistant Steel Sheet in Marine Atmosphere—W. H. Mutchler, presented by Leo J. Waldron.

Corrosion-Resistant Steel for Architectural and Structural Applications—H. A. Grove, presented by the author.

Atmospheric Corrosion Tests of Corrosion-Resistant Steel Wires—A. P. Jahn, presented by the author.

NINETEENTH SESSION—SYMPOSIUM ON FREEZING-AND-THAWING TESTS OF CONCRETE

THURSDAY, JUNE 27, 8.00 P.M.

SESSION CHAIRMAN: F. E. RICHART

The Symposium on Procedures for Making Freezing-and-Thawing Tests of Concrete was sponsored by Committee C-9 on Concrete and Concrete Aggregates to afford an opportunity for discussion of the procedures used by different investigators for evaluating the durability of concrete. The following general paper led off the discussion at this symposium:

Considerations Involved in the Making of Freezing-and-Thawing Tests on Concrete—M. O. Withey, presented by the author.

Following the lead paper by Prof. Withey, contributions from the following leading authorities were given:

C. E. Wuerpel, Central Concrete Laboratory, U. S. Army.
C. W. Allen, Ohio State Highway Testing and Research Lab.
W. H. Price, U. S. Bureau of Reclamation.
R. E. Davis, University of California
A. T. Goldbeck, National Crushed Stone Association.
F. V. Reagel, Missouri State Highway Commission.
Bailey Tremper, State Department of Highways, Washington.
R. C. Valore, Jr., National Bureau of Standards.
Stanton Walker, National Sand and Gravel Association.

TWENTIETH SESSION—CEMENT, CONCRETE, LIME, REFRACTORIES, MASONRY MATERIALS

FRIDAY, JUNE 28, 9.30 A.M.

SESSION CHAIRMAN: W. H. KLEIN

Committee C-1 on Cement:

Report presented by D. Wolochow, vice-chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Air-Entraining Portland Cement (C 175 – 46 T)

Adopted as Standard:

Chemical Analysis of Portland Cement (C 114 – 44 T), Sections 2 to 10 only

Adopted as Standard, Revisions in:

Spec. for Portland Cement (C 150 – 44)
Sampling Hydraulic Cement (C 183 – 44)

Accepted for Publication as Information Only:

Test for Sulfate Resistance of Portland Cements

Committee C-5 on Fire Tests of Materials and Construction:

Report presented by S. H. Ingberg, chairman, and the following actions taken:

Accepted as Tentative:

Test for Combustible Properties of Treated Wood by the Fire-Tube Method

Accepted as Tentative, Revisions in:

Methods of Fire Tests of Building Construction and Materials (C 19 – 41)

Adopted as Standard:

Test for Combustible Properties of Treated Wood by the Crib Test Method (C 160 – 41 T), as revised

Committee C-7 on Lime:

Report presented by W. C. Voss, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Hydrated Lime for Masonry Purposes
Spec. for Normal Finishing Hydrated Lime (Revision of C 6 – 44)
Spec. for Special Finishing Lime

Committee C-8 on Refractories:

Report presented, in the absence of the chairman, by R. E. Hess, Assistant Executive Secretary, and the following actions taken:

Accepted as Tentative, Revisions in:

Panel Test for Resistance to Thermal and Structural Spalling of Refractory Brick (C 38 – 45)
Panel Test for Resistance to Thermal and Structural Spalling of High Heat Duty Fireclay Brick (C 107 – 45)
Panel Test for Resistance to Thermal and Structural Spalling of Super Duty Fireclay Brick (C 122 – 45)
Spec. for Refractories for Malleable Iron Furnaces with Removable Bungs and for Annealing Ovens (C 63 – 41)

Panel Test for Resistance to Thermal and Structural Spalling of Fire Clay Plastic Refractories (C 180 – 45)
Spec. for Refractories for Heavy Duty Stationary Boiler Service (C 64 – 41)
Spec. for Refractories for Incinerators (C 106 – 41)
Spec. for Refractories for Moderate Duty Stationary Boiler Service (C 153 – 41)
Def. of Terms Relating to Refractories (C 71 – 45)
Test for True Specific Gravity of Burned Refractory Materials (C 135 – 40)
Spec. for Fireclay Plastic Refractories for Boiler and Incinerator Services (C 176 – 44)
Test for Workability Index of Fireclay Plastic Refractories (C 181 – 45)

Adopted as Standard:

Test for Permanent Linear Change Upon Reheating of Insulating Fire Brick (C 210 – 46)
Test for Sieve Analysis and Water Content of Refractory Materials (C 92 – 43 T)
Test for Combined Drying and Firing Shrinkage of Fireclay Plastic Refractories (C 179 – 43 T)
Symbols for Heat Transmission (C 108 – 44 T)

Adopted as Standard, Revisions in:

Testing Insulating Fire Brick (Compressive Strength, Flexural Strength, and Permanent Linear Change After Heating) (C 93 – 42)
Test for Permanent Linear Change After Reheating of Refractory Brick (C 113 – 36)
Test for Apparent Porosity, Water Absorption, Apparent Specific Gravity and Bulk Density of Burned Refractory Products (C 20 – 41)
Test for Pyrometric Cone Equivalent (P.C.E.) of Refractory Materials (C 24 – 42)
Def. of Terms Relating to Refractories (C 71 – 45)

Committee C-9 on Concrete and Concrete Aggregates:

Report presented by F. E. Richart, chairman, and the following actions taken:

Accepted as Tentative:

Def. of Terms Relating to Concrete and Concrete Aggregates (C 125 – 46 T)

Accepted as Tentative, Revisions in:

Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 – 44 T)[9]

Adopted as Standard, Revisions in:

Test for Structural Strength of Fine Aggregate Using Constant Water-Cement-Ratio Mortar (C 87 – 44)[9]
Spec. for Concrete Aggregates (C 33 – 44)[9]
Test for Sieve Analysis of Fine and Coarse Aggregates (C 136 – 39)[9]

Withdrawl of Standard:

Def. of Terms Relating to Concrete and Concrete Aggregates (C 125 – 44)

It was announced that Committee C-9 concurs with Committee D-4 on Road and Paving Materials in the revision of the Tentative Method of Test for Abrasion of Gravel by Use of the Deval Machine (D 289 – 42 T) and the immediate adoption of revisions in the Standard Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 – 44).

It was announced that the revisions under consideration in the Standard Specifications for Ready-Mixed Concrete (C 94 – 44), referred to in the report as preprinted, were being given further consideration in the committee and may subsequently be presented to the Society through the Standards Committee.

Sanford E. Thompson Award:

Mr. Richart then announced the selection of C. W. Muhlenbruch, Associate Professor of Civil Engineering, Carnegie Institute of Technology, to receive the Sanford E. Thompson Award for his paper on "The Effect of Repeated Loading on the Bond Strength of Concrete," published in the 1945 *Proceedings*. The Sanford E. Thompson Award was established in 1938 by Committee C-9 on Concrete and Concrete Aggregates as an annual token of recognition to the author or authors of a paper of outstanding merit on concrete and concrete aggregates presented at an annual meeting of the Society. The award is named in honor of the first chairman of the committee. The paper was considered noteworthy since it presented for the first time a series of tests which give accurate data on the effect of repeated loading, in a great range of cycles of load, on the bond strength of concrete.

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[9] The revisions under consideration, which were listed in the report as preprinted, were accepted subject to favorable letter ballot of Committee C-9, which ballot has been favorable and the results are given in the report of the committee, see p. 317.

Committee C-12 on Mortars for Unit Masonry:

Report presented, in the absence of the chairman, by H. W. Stauffer.

Committee C-13 on Concrete Pipe:

An oral progress report of this committee was presented by R. E. Hess, Assistant Executive Secretary.

Committee C-14 on Glass and Glass Products:

Report presented, in the absence of the chairman, by R. E. Hess, Assistant Executive Secretary.

Committee C-15 on Manufactured Masonry Units:

Report presented by D. E. Parsons, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Building Brick (Made from Clay or Shale) (C 62 – 44)
Spec. for Sand-Lime Building Brick (C 73 – 39)
Sampling and Testing Brick (C 67 – 44)

It was announced that the new Tentative Specifications for Structural Clay Facing Tile, which it had not been possible to include in the report as preprinted, would be presented to the Society through the Standards Committee subsequent to the Annual Meeting.

Committee C-16 on Thermal Insulating Materials:

Report presented, in the absence of the chairman, by R. E. Hess, Assistant Executive Secretary, and the following actions taken:

Accepted as Tentative:

Spec. for Structural Insulating Board Made from Vegetable Fibers
Testing Structural Insulating Board Made from Vegetable Fibers

Committee C-18 on Natural Building Stones:

Report presented, in the absence of the chairman, by R. E. Hess, Assistant Executive Secretary, and the following action taken:

Adopted as Standard:

Test for Compressive Strength of Natural Building Stone (C 170 – 41 T)

TWENTY-FIRST SESSION—NON-FERROUS METALS

FRIDAY, JUNE 28, 9.30 A.M.

SESSION CHAIRMAN: J. H. FOOTE

Committee B-1 on Wire for Electrical Conductors:

Report presented by J. H. Foote, chairman, and the following actions taken:

Adopted as Standard, Revisions in:

Spec. for Bronze Trolley Wire (B 9 – 39)
Spec. for Copper Trolley Wire (B 47 – 39)
Spec. for Figure-9 Deep-Section Grooved and Figure-8 Copper Trolley Wire for Industrial Haulage (B 116 – 40)
Spec. for Concentric-Lay-Stranded Copper Conductors, Hard, Medium-Hard, or Soft (B 8 – 44)
Spec. for Tinned Soft or Annealed Copper Wire for Electrical Conductors (B 33 – 45)

In each of the above standards editorial changes were submitted comprising the assigning of a number to each table in the specifications with appropriate titles and corresponding changes in the text to refer to the tables.

It was announced that the scope of Committee B-1 as given in the preprinted report had been further modified to read "specifications and methods of test for uninsulated electrical conductors composed of wire in various fabrications."

Committee B-2 on Non-Ferrous Metals and Alloys:

Report presented by E. E. Thum, chairman, and the following action taken:

Adopted as Standard, Revisions in:

Spec. for Special High-Grade Zinc (B 6 – 27)

It was announced that the Proposed Tentative Specifications for Fire-Refined Copper for Wrought Alloys, which were included in the preprinted report as information, would after some further minor changes be submitted to the Society through the Standards Committee for publication as tentative.

It was also stated that the committee had completed revisions in the Standard Specifications for White Metal Bearing Alloys (Known Commercially as "Babbitt Metal") (B 23 – 26) and in the Tentative Specifications for Soft Solder Metal (B 32 – 40 T) which would be presented to the Standards Committee subsequent to the Annual Meeting.

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys:

Report presented by H. S. Rawdon, chairman, and the following action taken:

Editorial Change Accepted in:

Method of Total Immersion Corrosion Testing of Non-Ferrous Metals (B 185 – 43 T)

Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys:

Report presented, in the absence of the chairman, by A. C. Hartshorne, and the following actions taken:

Adopted as Standard:

Spec. for Drawn or Rolled Alloy, 80 per cent Nickel, 20 per cent Chromium for Electrical Heating Elements (B 82 – 44 T)
Spec. for Drawn or Rolled Alloy, 60 per cent Nickel, 15 per cent Chromium, and Balance Iron, for Electrical Heating Elements (B 83 – 44 T)

It was announced that the title of Committee B-4 would, after approval by the Board of Directors, be changed to read "Electrical Heating, Resistance, and Related Alloys."

Committee B-5 on Copper and Copper Alloys, Cast and Wrought:

Report presented by G. H. Harnden, chairman, and the following actions taken:

Accepted as Tentative:

Rec. Practice for Preparing Tension Test Specimens for Copper-Base Alloy Castings

Accepted as Tentative, Revisions in:

Spec. for Cartridge Brass Sheet, Strip, Plate, Bar, and Disks (B 19 – 44 T)
Spec. for Naval Brass Rods, Bars, and Shapes (B 21 – 46 T)
Spec. for Bronze Castings for Turntables and Movable Bridges and for Bearing and Expansion Plates of Fixed Bridges (B 22 – 45 T)
Spec. for Brass Sheet and Strip (B 36 – 44 T)
Spec. for Seamless Copper Tubes (B 75 – 46 T)
Spec. for Leaded Brass Sheet and Strip (B 121 – 44 T)
Spec. for Copper-Nickel-Zinc and Copper-Nickel Alloy Sheet and Strip (B 122 – 46 T)
Spec. for Cartridge Brass Cartridge Case Cups (B 129 – 44 T)
Spec. for Gilding Metal Strip (B 130 – 44 T)
Spec. for Gilding Metal Bullet Jacket Cups (B 131 – 44 T)
Spec. for Leaded High-Strength Yellow Brass (Manganese Bronze) Sand Castings (B 132 – 44 T)
Spec. for Copper Rods, Bars, and Shapes (B 133 – 46 T)
Spec. for Miscellaneous Brass Tubes (B 135 – 46 T)
Spec. for Phosphor Bronze Rods, Bars, and Shapes (B 139 – 46 T)
Spec. for Tin-Bronze and Leaded Tin-Bronze Sand Castings (B 143 – 44 T)
Spec. for High-Leaded Tin-Bronze Sand Castings (B 144 – 45 T)
Spec. for Leaded Red Brass and Leaded Semi-Red Brass Sand Castings (B 145 – 45 T)
Spec. for Leaded Yellow Brass Sand Castings for General Purposes (B 146 – 45 T)
Spec. for High-Strength Yellow Brass (Manganese Bronze) and High-Strength Leaded Yellow Brass (Leaded Manganese Bronze) Sand Castings (B 147 – 44 T)
Spec. for Aluminum-Bronze Sand Castings (B 148 – 45 T)
Spec. for Leaded Nickel-Brass (Leaded Nickel-Silver) and Leaded Nickel-Bronze (Leaded Nickel-Silver) Sand Castings (B 149 – 44 T)
Spec. for Aluminum Bronze Rods, Bars, and Shapes (B 150 – 46 T)
Spec. for Copper-Nickel-Zinc Alloy Rod, Bar, and Wire (B 151 – 46 T)
Spec. for Copper Sheet, Strip, and Plate (B 152 – 45 T)
Spec. for Phosphor Bronze Wire (B 159 – 46 T)
Spec. for Aluminum Bronze Sheet and Strip (B 169 – 44 T)
Spec. for Copper Bus Bars, Rods and Shapes (B 187 – 45 T)
Spec. for Copper Bus Pipes and Tubes (B 188 – 45 T)
Spec. for Beryllium-Copper Alloy Strip (B 194 – 46 T)
Spec. for Beryllium-Copper Alloy Strip, Special Grade (B 195 – 45 T)
Spec. for Beryllium-Copper Alloy Rod and Bar (B 196 – 46 T)
Spec. for Beryllium-Copper Alloy Wire (B 197 – 46 T)
Spec. for Silicon-Bronze and Silicon-Brass Sand Castings (B 198 – 45 T)

Spec. for Copper-Nickel-Zinc Alloy Wire (B 206 - 46 T)

Adopted as Standard, Revisions in:

Spec. for Copper Plates for Locomotive Fireboxes (B 11 - 41)
Spec. for Copper Bars for Locomotive Staybolts (B 12 - 45)
Spec. for Free-Cutting Brass Rod and Bar for Use in Screw Machines (B 16 - 45)
Spec. for Copper Pipe, Standard Sizes (B 42 - 45)
Spec. for Brass Pipe, Standard Sizes (B 43 - 45)
Spec. for Phosphor Copper (B 52 - 43)
Spec. for Silicon Copper (B 53 - 43)
Spec. for Steam or Valve Bronze Castings (B 61 - 44)
Spec. for Composition Brass or Ounce Metal Castings (B 62 - 44)
Spec. for Bronze Castings in the Rough for Locomotive Wearing Parts (B 66 - 44)
Spec. for Car and Tender Journal Bearings, Lined (B 67 - 44)
Spec. for Seamless Copper Tubing, Bright Annealed (B 68 - 45)
Spec. for Copper Water Tube (B 88 - 45)
Spec. for Copper-Silicon Alloy Plate and Sheet for Pressure Vessels (B 96 - 42)
Spec. for Copper-Silicon Alloy Sheet and Strip for General Purposes (B 97 - 44)
Spec. for Copper-Silicon Alloy Rods, Bars and Shapes (B 98 - 45)
Spec. for Copper-Silicon Alloy Wire for General Purposes (B 99 - 45)
Spec. for Rolled Copper-Alloy Bearing and Expansion Plates for Bridge and Other Structural Uses (B 100 - 44)
Spec. for Phosphor Bronze Sheet and Strip (B 103 - 44)
Spec. for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 - 45)
Spec. for Copper-Base Alloy Forging Rods, Bars and Shapes (B 124 - 45)
Spec. for Brass Wire (B 134 - 45)
Spec. for Manganese Bronze Rods, Bars, and Shapes (B 138 - 45)
Spec. for Leaded Red Brass (Hardware Bronze) Rods, Bars, and Shapes (B 140 - 45)
Spec. for Copper-Alloy Condenser Tube Plates (B 171 - 45)

Committee B-6 on Die-Cast Metals and Alloys:

Report presented by J. R Townsend, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Aluminum-Base Alloy Die Castings (B 85 - 44 T),[10] comprising the addition of two new aluminum-base alloys SG2 and SG3, requiring the following changes in these specifications:

Section 1.—Change the second sentence to refer to nine alloys and add references to alloys SG2 and SG3.

Table I.—Add the following requirements on chemical composition for the two new alloys:

	Alloy SG2	Alloy SG3
Copper, max., per cent	0.6	0.6
Iron, max., per cent	1.3	2.0
Silicon, per cent	9.0 to 10.0	9.0 to 10.0
Manganese, max., per cent	0.3	0.3
Magnesium, per cent	0.4 to 0.6	0.4 to 0.6
Zinc, max., per cent	0.5	0.5
Nickel, max., per cent	0.5	0.5
Tin, max., per cent	0.1	0.1
Other constituents, except aluminum, max., (total) per cent	0.2	0.2
Aluminum, per cent	remainder	remainder

Also change footnote *b* under Table I to read as follows: "Alloys S4, S5, SC2, SC7 and SG3 are suitable for the production of castings by either the gooseneck or the cold-chamber process. Castings of alloys S9, SC5, SC6 and SG2 may be made only in cold-chamber machines."

Appendix.—Add the following data on mechanical properties of the two new alloys:

	Alloy SG2	Alloy SG3
Tensile strength (round specimens), psi	43 000	43 000
Elongation in 2 in. (round specimens), per cent	5	3
Charpy Impact (square specimens), ft-lb.	4.2	2.7

Adopted as Standard, Revisions in:

Spec. for Zinc-Base Alloy Die Castings (B 86 - 43),[10] comprising the following changes:

New Section.—Add the following new Section 6, renumbering the remaining sections accordingly:

6. *Chemical Analysis.*—The chemical analyses of the die castings shall be made in accordance with the Tentative Methods of Spectrochemical Analysis of Zinc-Alloy

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[10] This recommendation, not preprinted, was accepted by unanimous consent, subject to favorable letter ballot of Committee B-6, which ballot has been favorable and the results are given in the report of the committee, see p. 224.

Die Castings for Minor Constituents and Impurities (A.S.T.M. Designation: E 27) and with the Tentative Methods of Chemical Analysis of Zinc-Base Die-Casting Alloys (A.S.T.M. Designation: E 47) for the major constituents.

Sections 7 and 8.—Omit these sections on stability and expansion tests, respectively.

Withdrawal of Tentative:

Spec. for Special Quality Zinc-Base Alloy Die Castings (B 186 – 42 T)

The chairman also submitted the following two reports for publication in the Proceedings as appendices to the Report of Committee B-6, subject to acceptance by the Committee on Papers and Publications:

Report on an Investigation of Aluminum Die-Casting Alloys Nos. IVa, Va and V.

Report on Creep Tests on Tin- and Lead-Base Die Castings.

It was announced that Committee B-6 would recommend to the Standards Committee the withdrawal of the Emergency Specifications for Special Quality Aluminum-Base Alloy Die Castings (ES – 29), and the Emergency Specifications for Special Quality, Magnesium-Base Alloy Die Castings (ES – 40).

It was also announced that the committee recommended the continuation as tentative of the Specifications for Magnesium-Base Alloy Die Castings (B 94 – 44 T) and the Specifications for Copper-Base (Brass) Alloy Die Castings (B 176 – 42 T).

Committee B-7 on Light Metals and Alloys, Cast and Wrought:

Report presented by D. L. Colwell, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Aluminum and Aluminum-Alloy Sheet and Plate, with the following revisions:[11]

Section 4 (a).—In the first sentence insert the words "of non-clad sheet" after "chemical analysis." Also add the following as a new Paragraph (*c*):

(*c*) Samples for determining the analysis of the core in clad materials may be taken in the manner described in Paragraph (*b*) but either the effect of the composition of the cladding shall be considered in calculating the results or the cladding shall be substantially removed from the sheet or plate before the final sample for analysis is prepared.

NOTE.—The analysis of clad material in the finished state is a difficult matter because of the relatively thin layer of cladding present and the effect of diffusion between the core and cladding during heat treatment. A reasonably satisfactory determination of the core composition can be made if the cladding is substantially removed from the sheet before analysis. Determination of the cladding analysis is considerably more difficult not only because of its thinness, but also because diffusion causes an indeterminate change in the composition. That the correct cladding alloy has been used, however, can usually be determined reasonably well by a combination of metallographic examination and spectrographic analysis of the surface at several widely separated points.

Table I.—Add the following chemical composition requirements for alloy 61S:

		Alloy 61S
Copper, per cent	min.	0.15
	max.	0.40
Iron, per cent, max.		0.70
Silicon, per cent	min.	0.4
	max.	0.8
Manganese, per cent	min.	..
	max.	0.15
Magnesium, per cent	min.	0.8
	max	1.2
Zinc, per cent	min.	...
	max.	0.20
Chromium, per cent	min.	0.15
	max.	0.35
Titanium, per cent, max.		0.15
Other elements, per cent	each, max.	0.05
	total, max.	0.15
Aluminum, per cent		remainder

Also add a reference letter *d* after the word "cladding" in the first column of the table referring to the following footnote:

[d] Composition of cladding alloy as applied during course of manufacture. Sample from finished sheet or plate not required to conform with these limits.

Table II.—To correct typographical errors in the specifications as preprinted, change the tensile strength value for alloy 2S from "15,000" to "15,500" for the annealed temper in the thickness range 0.250 to 3.000 in.; also for alloy 2S change the elongation in 2 in. from "18" to "23" for the annealed temperature in the thickness range 0.250 to 3.000 in. For alloy 52S, change the tensile strength from "34,000" to "31,000" for annealed temper in

the same thickness range; also change elongation in 2 in. from "12" to "18." Also, in the as-rolled temper of alloy 52S, change the tensile strength from "34,000" to "28,000" for thickness range 0.250 to 0.500 in. and from "31,000" to "24,000" for thickness range 0.501 to 2.000 in. Also change the elongation from "9" to "12." For alloy R303 interchange the values for yield strength of 65,000 and 60,000 respectively.

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[11] This recommendation, not preprinted, was accepted by unanimous consent subject to favorable letter ballot of Committee B-7, which ballot has been favorable and the results are given in the report of the committee, see p. 262.

New Sections.—Add the following new Sections 11 and 12, renumbering the present sections accordingly:

11. *Retests.*—If any tension specimen fails to conform to the requirements prescribed in Table II, two additional specimens shall be selected and tested. If either of these specimens fails to conform to the applicable requirements, the material may be rejected. If, however, the failure of the specimens to conform to the requirements is the result of an inadequate thermal treatment, the material may be reannealed or reheat treated, as applicable, and resampled in accordance with Section 9. Only one such reworking of the material shall be permitted.

12. *Cladding Thickness* (*a*).—The aluminum or aluminum-alloy plates which are bonded to the alloy ingot or slab preparatory to rolling to the specified thickness of plate or sheet shall be of the composition shown in Table I and shall each have a thickness not less than that shown as follows for the alloy specified:

Alloy	Total Composite Thickness of Finished Sheet,[a] in.	Minimum Thickness of Cladding, in.
Clad CG1	0.063 and under	0.05 T[a]
	0.064 and over	0.025 T[a]
Clad CS41	0.024 and under	0.10 T[a]
	0.025 to 0.039	0.075 T[a]
	0.040 to 0.101	0.05 T[a]
	0.102 and over	0.025 T[a]
Clad ZG42 Clad ZG43	all	0.04 T[a]

[a] "T" is the total composite thickness in inches.

(*b*) When the thickness of the cladding is to be determined on finished material, not less than three transverse samples approximately $\frac{3}{4}$ in. in length shall be mounted to expose an edge perpendicular to the plane of the sample and polished for microscopic examination. Using a magnification of 100 diameters, the maximum and minimum coating thickness shall be measured in each of five fields approximately 0.1 in. apart along both sides of the cross-section. The average of the ten thickness measurements on each side is the average coating thickness and shall be not less than 80 per cent of the thickness calculated from the above requirements.

Section 14.—Renumber as Section 16 and, to correct an omission, add the following: "be rejected. If rejected, the manufacturer's responsibility shall be limited to replacing the rejected material without charge to the purchaser. The full weight of the rejected material shall be returned to the manufacturer."

Spec. for Aluminum-Alloy Drawn Seamless Tubing

Spec. for Aluminum-Alloy Bars, Rods and Wire

Accepted as Tentative, Revisions in:

Spec. for Magnesium-Base Alloy Sheet (B 90 – 45 T)

Spec. for Aluminum-Manganese Alloy Sheet and Plate for Use in Welded Pressure Vessels (B 126 – 44 T)

Spec. for Aluminum Sheet and Plate for Use in Welded Pressure Vessels (B 178 – 44 T)

Adopted as Standard, Revisions in:

Spec. for Aluminum Ingots for Remelting (B 24 – 44)

Withdrawal of Tentatives:

Spec. for Aluminum-Alloy (Duralumin) Sheet and Plate (B 78 – 44 T)

Spec. for Aluminum Sheet and Plate (B 25 – 44 T)

Spec. for Aluminum-Manganese Alloy Sheet and Plate (B 79 – 44 T)

Spec. for Aluminum-Alloy (Duralumin) Bars, Rods, Wire and Shapes (B 89 – 44 T)

Spec. for Aluminum-Magnesium-Chromium Alloy Sheet and Plate (B 109 – 44 T)

Committee B-8 on Electrodeposited Metallic Coatings:

Report presented, in the absence of the chairman, by A. W. Tracy.

Committee B-9 on Metal Powders and Metal Powder Products:

Reported presented by W. A. Reich, chairman, and the following actions taken:

Accepted as Tentative:

Test for Flow Rate of Metal Powders

Test for Sieve Analysis of Granular Metal Powders

Test for Apparent Density of Metal Powders

Method of Sampling Finished Lots of Metal Powders

TWENTY-SECOND SESSION—MISCELLANEOUS MATERIALS—TECHNICAL PAPERS ON INSULATING MATERIALS

FRIDAY, JUNE 28, 9.30 A.M.

SESSION CHAIRMAN: VICE-PRESIDENT ARTHUR W. CARPENTER

Committee D-19 on Water for Industrial Uses:

Report presented by Max Hecht, chairman, and the following actions taken:

Accepted as Tentative:

Test for Dissolved Oxygen in Industrial Waters

Rec. Practice for Field Sampling of Water-Formed Deposits

Accepted as Tentative, Revisions in:

Test for Tendency of Boiler Water to Cause Embrittlement Cracking of Steel (D 807 – 44 T)

Adopted as Standard, Revisions in:

Method of Reporting Results of Analysis of Industrial Waters (D 596 – 41)

Joint Research Committee on Boiler Feed-water Studies:

An oral progress report presented, in the absence of the chairman, by Max Hecht.

Committee D-5 on Coal and Coke:

Report presented by A. C. Fieldner, chairman, and the following actions taken:

Adopted as Standard:

Test for Free-Swelling Index of Coal (D 720 – 43 T)[12]

Sampling Coals Classed According to Ash Content (D 492 – 43 T),[12] with the following minor revisions:

Section 4 (a).—Change the second sentence to read as follows by the addition of the italicized word and the omission of the word in brackets: "This procedure is *intended,* [designed] for an accuracy such that if a large number of samples were taken from a single lot of coal, the test results in 95 out of 100 cases would fall within plus or minus 10 per cent of the average ash content of these samples." Also at the end of this sentence add a reference to the following footnote: "It is recognized that this accuracy specification may not be obtained for all size groups and ranges of ash shown in Table I. Further sampling investigations are in progress to obtain additional data and may result in revision of the accuracy specification."

Adopted as Standard, Revisions in:

Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 – 44)[12], comprising the inclusion of the tentative revision[13] issued in June, 1945.

Committee D-3 on Gaseous Fuels:

Report presented by A. W. Gauger, chairman. It was announced that the proposed Tentative Method of Test for Calorific Value of Gaseous Fuels by the Water-Flow Calorimeter, which it had not been possible to include in the report as preprinted, had been approved by letter ballot of Committee D-3 and would be presented for acceptance by the Standards Committee subsequent to the annual meeting.

Committee D-6 on Paper and Paper Products:

Report presented by G. H. Harnden, secretary, and the following actions taken:

Accepted as Tentative, Revisions in:

Qualitative Examination of Mineral Filler and Mineral Coating of Paper (D 686 – 43 T)

Test for Resistance of Paper to Passage of Air (D 726 – 43 T)

Test for Ply Adhesion of Paper or Vulcanized Fibre (D 825 – 45 T)

Adopted as Standard:

Test for Bursting Strength of Paper (D 774 – 44 T

Test for Effect of Heating on Folding Endurance of Paper (D 776 – 44 T)

Test for Flammability of Treated Paper and Paperboard (D 777 – 44 T)

Test for Hydrogen Ion Concentration (pH) of Paper Extracts (D 778 – 44 T)

Test for Time of Penetration by Water of Sized Paper and Paper Products (Dry Indicator Method) (D 779 – 44 T)

Test for Printing Ink Permeation of Paper (Castor Oil Test) (D 780 – 44 T)

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[12] These recommendations, not preprinted, were accepted by unanimous consent, subject to favorable letter ballot of Committee D-5, which ballot has been favorable and the results are given in the report of the committee, see p. 373.

[13] 1945 Supplement to Book of A.S.T.M. Standards, Part III, p. 485.

Adopted as Standard, Revisions in:

Test for Resin in Paper (D 549 – 41)

Accepted for Publication as Information Only:

Test for the Bacteriological Examination of Paper and Paperboard

The committee withdrew from the report as preprinted the recommendation for adoption as standard of the Tentative Method of Test for Adhesiveness of Gummed Tape (D 773 – 44 T).

Committee D-7 on Wood:

Report presented by Hermann von Schrenk, chairman.

Committee D-9 on Electrical Insulating Materials:

Report presented by W. A. Zinzow, secretary, and the following actions taken:

Accepted as Tentative:

Testing Nonrigid Polyvinyl Tubing
Test for Dielectric Strength of Insulating Oil of Petroleum Origin
Test for Inorganic Chlorides and Sulfates in Insulating Oils
Spec. for Communication and Signal Pin-Type Lime-Glass Insulators[14] (formerly Emergency Standard ES – 41)

Accepted as Tentative, Revisions in:

Testing Molded Materials Used for Electrical Insulation (D 48 – 45 T)
Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (D 150 – 45 T), with additional editorial changes
Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 – 45 T)
Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (D 295 – 44 T)
Method for Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 45 T), jointly with Committee D–20 on Plastics, with Notes 1 and 2 in Section 3 reworded to read as follows:

Note 1.—Standard Procedure is generally satisfactory and recommended for use unless functional procedures are specified.

Note 2.—Functional Procedure A is recommended for use wherever the specific effects of drying are to be determined.

Spec. for Laminated Thermosetting Materials (D 709 – 44 T), subject to concurrence of Committee D-20.
Spec. for Low and Medium Voltage Pin-Type Lime-Glass Insulators (D 730 – 43 T)
Testing Varnishes Used in Electrical Insulation (D 115 – 41)

Adopted as Standard:

Test for Power Factor and Dielectric Constant of Natural Mica (D 351 – 42 T), with minor editorial changes.[14]

Adopted as Standard, Revisions in:

Test for Insulation Resistance of Electrical Insulating Materials (D 257 – 38), with additional editorial changes.
Testing Pin-Type Lime-Glass Insulators (D 468 – 42)
Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 – 43)
Testing Tubes Used in Electrical Insulation (D 348 – 42)
Testing Laminated Rods Used in Electrical Insulation (D 349 – 42)
Test for Grading and Classification of Natural Mica (D 351 – 38), with minor editorial changes
Test for Acetone Extraction of Phenolic Molded or Laminated Products (D 494 – 41),[15] jointly with Committee D-20.

Committee D-10 on Shipping Containers:

Report presented, in the absence of the chairman, by L. J. Markwardt, and the following action taken:

Accepted as Tentative:

Method of Incline-Impact Test for Shipping Containers

Committee D-11 on Rubber and Rubber-Like Materials:

Report presented by Simon Collier, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Test for Compression Set of Vulcanized Rubber (D 395 – 40 T)
Testing Cellular Rubber Products (D 552 – 46 T)
Testing Asphalt Composition Battery Containers (D 639 – 43 T)

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[14] This recommendation, not preprinted, was accepted by unanimous consent, subject to favorable letter ballot of Committee D-9, which ballot has been favorable and the results are given in the report of the committee, see p. 394.

[15] This recommendation was accepted by unanimous consent, subject to favorable letter ballot of Committee D-9, which ballot has been favorable and the results are given in the report of the Committee, see p. 394

Test for Indentation of Rubber by Means of the Durometer (D 676 – 44 T)
Spec. for Rubber and Synthetic Rubber Compounds for Automotive and Aeronautical Applications (D 735 – 43 T)
Test for Low-Temperature Brittleness of Rubber and Rubber-Like Materials (D 736 – 43 T)
Spec. for GR-M Polychloroprene Sheath Compound for Electrical Insulated Cords and Cables (D 752 – 44 T)
Spec. for Cellular Rubber Products (D 798 – 46 T)
Test for Permeability of Vulcanized Rubber or Synthetic Elastomers to Volatile Liquids (D 814 – 44 T)
Testing Rubber Adhesives (D 816 – 44 T)
Rec. Practice for Conditioning of Rubber and Plastic Materials for Low-Temperature Testing (D 832 – 45 T)
Spec. for Friction Tape for General Use for Electrical Purposes (D 69 – 38)
Spec. for Rubber Insulating Tape (D 119 – 38)

Accepted as Tentative:

Spec. for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (D 27 – 41),[16] this standard being reverted to tentative since the committee plans to incorporate the Emergency Alternate Provisions EA – D 27a.
Testing Rubber Insulated Wire and Cable (D 470 – 41),[16] reverted to tentative for the incorporation of the Emergency Alternate Provisions EA – D 470, and also the changes recommended for immediate adoption in the report as preprinted.

Adopted as Standard:

Test for Compression-Deflection Characteristics of Vulcanized Rubber (D 575 – 40 T)
Test for Young's Modulus in Flexure of Natural and Synthetic Elastomers at Normal and Subnormal Temperatures (D 797 – 44 T)
Testing Compressed Asbestos Sheet Packing (D 733 – 43 T)

Adopted as Standard, Revisions in:

Test for Abrasion Resistance of Rubber Compounds (D 394 – 40)
Spec. for Insulated Wire and Cable: Heat-Resisting Rubber Compound (D 469 – 41)

The recommendation appearing in the report as preprinted for the immediate adoption of revisions in the Standard Method of Test for Tear Resistance of Vulcanized Rubber (D 624 – 44) was withdrawn by the committee.

Committee D-12 on Soaps and Other Detergents:

Report presented, in the absence of the chairman, by the chair, and the following actions taken:

Accepted as Tentative, Revisions in:

Def. of Terms Relating to Soaps and Other Detergents (D 459 – 45 T)
Spec. for Milled Soap (D 455 – 39)
Spec. for Chip Soap (D 496 – 39)
Spec. for Powdered Soap (D 498 – 39)
Spec. for White Floating Toilet Soap (D 499 – 39)
Spec. for Palm Oil Solid Soap (D 535 – 41)
Spec. for Palm Oil Chip Soap (D 536 – 42)
Spec. for Olive Oil Solid Soap (D 592 – 42)
Spec. for Olive Oil Chip Soap (D 630 – 42)
Spec. for Chip Soap with Rosin (D 690 – 44)

Adopted as Standard:

Chemical Analysis of Soaps Containing Synthetic Detergents (D 820 – 45 T), as revised

Adopted as Standard, Revisions in:

Sampling and Chemical Analysis of Soaps (D 460 – 44)
Sampling and Chemical Analysis of Special Detergents (D 501 – 44)

Reaffirmation of Standards:

Spec. for Caustic Soda (D 456 – 39)
Spec. for Modified Soda (D 457 – 39)
Spec. for Soda Ash (D 458 – 39)
Spec. for Ordinary Bar Soap (D 497 – 39)

Committee D-13 on Textile Materials:

Report presented, in the absence of the chairman, by D. C. Scott, Jr., and the following cations taken:

Accepted as Tentative:

Testing and Tolerances for Rayon Tire Cord
Test for Compatability of Glass Yarn with Insulating Varnish

Accepted as Tentative, Revisions in:

Spec. for Woven Asbestos Cloth (D 677 – 42 T)
Testing and Tolerances for Glass Yarn (D 578 – 44 T)
Quantitative Analysis of Textiles (D 629 – 42 T)

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[16] These recommendations, not preprinted, were accepted by unanimous consent, subject to favorable letter ballot of Committee D-11, which ballot has been favorable and the results are given in the report of the Committee, see p. 419.

Def. of Terms Relating to Textile Materials (D 123 - 45 T)
Rec. Practice for a Universal System of Yarn Numbering (D 861 - 45 T)
Test for Fineness of Wool (D 419 - 44)
Spec. and Test for Fineness of Wool Tops (D 472 - 41)
Test for Hard Scoured Wool in Wool in the Grease (Laboratory Scale Operations) (D 584 - 43)
Spec. for Textile Testing Machines (D 76 - 45)
Testing and Tolerances for Tire Cord, Woven and on Cones (D 179 - 42)
Testing and Tolerances for Continuous Filament Rayon Yarns (D 258 - 44)[17]

Adopted as Standard:

Testing and Tolerances for Rope (Leaf and Bast Fibers) (D 738 - 45 T)
Testing and Tolerances for Spun, Twisted, or Braided Products Made from Flax, Hemp, Ramie, or Mixtures Thereof (D 739 - 45 T)
Test for Air Permeability of Textile Fabrics (D 737 - 43 T)

Adopted as Standard, Revisions in:

Testing and Tolerances for Knit Goods (D 231 - 39)

Reaffirmation of Standards:

Spec. and Test for Osnaburg Cement Sacks (D 205 - 39)
Spec. and Test for Cotton Goods for Rubber and Pyroxylin Coating (D 334 - 40)
Testing and Tolerances for Certain Carded Cotton Gray Goods (D 433 - 39)
Spec. for 0.007-in. Cotton Tape for Electrical Purposes (D 335 - 36)
Test for Strength of Rayon Woven Fabric When Wet (D 415 - 38)
Test for Maximum Residual Shrinkage of Silk and Rayon Woven Fabrics (D 416 - 39)
Test for Fiber Length of Wool (D 519 - 40)
Spec. for Terry (Turkish) Toweling (D 505 - 40)

Committee D-14 on Adhesives:

Report presented by Henry Grinsfelder, secretary. It was announced that the following eight new tentative methods had been approved by the committee and would be submitted for acceptance by the Standards Committee subsequent to the annual meeting:

Testing Strength Properties of Adhesives
Testing Strength Properties of Adhesives in Plywood Type Construction in Shear by Tension Loading
Test for Resistance of Adhesive Bonds to Chemical Reagents
Method for Determining Applied Weight per Unit Area of Water Soluble Adhesive
Method for Determining Applied Weight per Unit Area of Adhesive Containing Volatile Solvents.
Method for Determining Peel or Stripping Strength of Adhesives
Testing Strength Properties of Adhesives, Tensile Strength
Rec. Practice for Determining Effect of Artificial and Natural Light on the Permanence of Adhesives

Committee D-16 on Industrial Aromatic Hydrocarbons:

Report presented by J. M. Weiss, chairman, and the following actions taken:

Accepted as Tentative:

Test for Specific Gravity of Industrial Aromatic Hydrocarbons

Accepted as Tentative, Revisions in:

Spec. for Industrial Grade Benzene (D 836 - 45 T),[18] with an addition to Section 2 of the following requirement for acidity: "no free acid, that is, no evidence of acidity," with appropriate changes in Section 3 referring to the applicable test method.

Editorial Changes Accepted in:

Test for Acid Wash Color of Benzene, Toluene, Xylenes and Similar Industrial Aromatic Hydrocarbons (D 848 - 45 T)
Test for Distillation of Industrial Aromatic Hydrocarbons (D 850 - 45 T)

Committee D-17 on Naval Stores:

Report presented by V. E. Grotlisch, chairman, and the following actions taken:

Accepted as Tentative:

Test for Water in Liquid Naval Stores
Test for Volatile Oil in Rosin, with a revision in Section 2 (*a*) changing "300-ml. pyrex round-bottom flask" to read "500-ml. round-bottom flask" and omit the note; also the addition of the following explanatory note to Section 2 (*b*):

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[17] This recommendation appeared incorrectly in the report as preprinted as a revision of standard for immediate adoption.

[18] This recommendation, not preprinted, was accepted by unanimous consent, subject to favorable letter ballot of Committee D-16, which ballot has been favorable and the results are given in the report of the committee, see p. 442.

NOTE.—This trap is a modification of the original Clevenger trap used to recover oils lighter than water. In that trap the oil and water condensate drop directly into the graduated part of the trap. Due to the viscous character, the density and surface tension of the oils recovered from rosin, cylindrical columns of oil were formed below the surface of the water in the graduated section of the trap, which were not penetrated by the water condensed immediately thereafter. This resulted in alternate columns of oil and water in the graduated section. These were returned to the distilling flask in the same order as they occurred, through the side arm of the trap. As this condition continued indefinitely, it was impossible to completely remove all the oil from the rosin. By raising the opening of the side arm of the trap to the position shown, to bring the surface of the liquid into the wide part of the trap above the narrow graduated section, the oil is collected in a thinner film which can be readily penetrated by the droplets of water falling from the end of the condenser, and only the water is thus collected in the narrow graduated section. At the end of the test, the oil is slowly brought down into the graduated section and its volume read. The system or apparatus loss amounted to not more than 0.1 ml. of oil.

Accepted as Tentative, Revisions in:

Def. of Terms Relating to Naval Stores and Related Products (D 804 - 45 T), except that the definition of the term "Niggre" was withdrawn by the committee.

Sampling and Testing Pine Tars and Pine-Tar Oils (D 856 - 45 T)

Sampling and Testing Dipentene (D 801 - 44 T) with the addition of the following note at the beginning of Section 15 (*a*):

> NOTE.—If desired, the aniline and mixed aniline points of dipentene may be determined in accordance with the Tentative Method of Test for Aniline Point and Mixed Aniline Point of Petroleum Products (A.S.T.M. Designation: D 611).

Sampling and Testing Pine Oil (D 802 - 44 T)

The recommendation for publication as tentative of the Method of Test for Petroleum Spirits (Naphtha) Insoluble Matter in Rosin, which appeared in the preprinted report, was withdrawn by the committee.

Committee D-20 on Plastics:

Report presented by A. J. Warner, secretary-elect, and the following actions taken:

Accepted as Tentative:

Method for Estimating Blocking of Plastic Sheets

Test for Tensile Properties of Thin Plastic Sheets and Films

Test for Deviation of Line of Sight Through Transparent Plastics[19]

Def. of Terms Relating to Plastics, comprising the terms plastic, as an adjective, and pseudostable.

Accepted as Tentative, Revisions in:

Spec. for Cellulose Acetate Molding Compounds (D 706 - 44 T)

Spec. for Cellulose Acetate Butyrate Molding Compounds (D 707 - 44 T)

Spec. for Nonrigid Vinyl Chloride-Acetate Resin Plastics (D 742 - 44 T)

Spec. for Ethyl Cellulose Molding Compounds (D 787 - 44 T)

Spec. for Cellulose Nitrate Molding Compounds (D 701 - 44 T)

Test for Impact Resistance of Plastics and Electrical Insulating Materials (D 256 - 43 T), subject to concurrence of Committee D-9 on Electrical Insulating Materials.

Test for Tensile Properties of Plastics (D 638 - 44 T)

Test for Resistance of Plastics to Accelerated Service Conditions (D 756 - 44 T)

Method of Conditioning Plastics (D 618 - 45 T), jointly with Committee D-9

Rec. Practice for Long-Time Tension Tests of Plastics (D 674 - 42 T),[20] changing this to a method of test

Adopted as Standard:

Spec. for Cast Methacrylate Plastic Sheets, Rods, Tubes, and Shapes (D 702 - 43 T)

Spec. for Urea-Formaldehyde Molding Compounds (D 705 - 43 T)

Test for Shear Strength of Plastics (D 732 - 43 T)

Adopted as Standard, Revisions in:

Test for Acetone Extraction (D 494 - 41), jointly with Committee D-9

It was announced that the committee had withdrawn from the report as preprinted two recommendations; namely, the proposed Method of Test for Apparent Density and Bulk Factor of Nonpouring Molding Powders, and the adoption as standard of the Tentative Method of Test for Stiffness in Flexure of Nonrigid Plastics (D 747 - 43 T).

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[19] This recommendation, not preprinted, was accepted by unanimous consent, subject to favorable letter ballot of Committee D-20, which ballot has been favorable and the results are given in the report of the committee, see p. 462.

[20] This recommendation appeared incorrectly in the report as preprinted as a tentative recommended for adoption as standard.

It was also announced that the proposed Tentative Definition of the Term Plastic as a noun had been presented prior to the annual meeting to the Standards Committee.

Papers:

The following papers were presented:

Water Determination in New and Used Insulating Oils by Doble Method—Frank C. Doble, presented by the author.

Electrical Quality Classification of Raw Mica by a Rapid, Direct-Reading Test Method—K. G. Coutlee, presented from manuscript by the author, including a motion picture film which showed the apparatus and illustrated the application of the test method in the classification of mica.

TWENTY-THIRD SESSION—TECHNICAL PAPERS ON PLASTICS AND TIMBER

FRIDAY, JUNE 28, 2 P.M.

SESSION CHAIRMAN: J. H. ADAMS

Tension and Torsion Creep Properties of Cloth Laminates[21]—Joseph Marin, presented by title only.

Effect of Weathering on Insulation Resistance of Plastics—Leslie H. Campbell, Arthur H. Falk, and Robert Burns, presented from manuscript by Mr. Campbell.

Effect of Temperature and Humidity on Mechanical Properties of Molded Cellulose Acetate Plastics—W. E. Welch, R. F. Hayes, T. S. Carswell, and H. K. Nason, presented by Mr. Carl Frosch.

Methods for Determining the Specific Gravity of Wood and Wood-Base Materials—L. J. Markwardt and B. H. Paul, presented from manuscript by Mr. Markwardt.

TWENTY-FOURTH SESSION—TECHNICAL PAPERS ON NON-FERROUS METALS

FRIDAY, JUNE 28, 2.00 P.M.

SESSION CHAIRMAN: R. L. TEMPLIN

The Notch Sensitivity in Static and Impact Loading of Some Magnesium-Base and Aluminum-Base Alloys—J. P. Doan and J. C. McDonald, presented by Mr. McDonald.

Atmospheric and Indoor Aging Studies on Soem Aluminum and Zinc-Base Die-Casting Alloys—G. R. Gohn and Lucille E. Menges, presented by Mr. Gohn.

Evaluation of the Forming Properties in Bending of Five Commercial Sheet Aluminum Alloys—G. R. Gohn and S. M. Arnold, presented by Mr. Arnold.

Creep Tests on Some Extruded Lead and Lead Alloy Sleeves and Tapes—G. R. Gohn, S. M. Arnold, and G. M. Bouton, presented by Mr. Gohn.

A Permeameter for Metals Used in Cathode-Ray and Television Tubes—H. J. Evans, presented from manuscript by the author.

Compressive Stress-Strain Properties of Some Aircraft Materials—P. E. Sandorff and R. K. Dillon, presented from manuscript by Mr. F. R. Shanley.

TWENTY-FIFTH SESSION—BITUMINOUS MATERIALS

FRIDAY, JUNE 28, 2.00 P.M.

SESSION CHAIRMAN: J. S. MILLER

Committee D-4 on Road and Paving Materials:

Report presented by Shreve Clark, chairman, and the following actions taken:

Accepted as Tentative:

Test for Sulfonation Index of Road Tars

Accepted as Tentative, Revisions in:

Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials (D 75 - 42 T)

Sampling Bituminous Materials (D 140 - 41 T), jointly with Committee D-8 on Bituminous Waterproofing and Roofing Materials

Spec. for Calcium Chloride (D 98 - 34)

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[21] ASTM BULLETIN, No. 143, December, 1946.

Chemical Analysis of Calcium Chloride (D 345 - 34)

Adopted as Standard:

Spec. for Cut-Back Asphalt (Rapid Curing Type) (D 597 - 40 T)

Spec. for Cut-Back Asphalt (Medium Curing Type) (D 598 - 40 T)

Test for Abrasion of Gravel by Use of the Deval Machine (D 289 - 42 T), as revised, subject to concurrence of Committee C-9 on Concrete and Concrete Aggregates

Adopted as Standard, Revisions in:

Spec. for Slow-Setting Emulsified Asphalt (D 631 - 43)

Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 - 44), subject to concurrence of Committee C-9

Test for Sieve Analysis of Fine and Coarse Aggregates (C 136 - 39)

Def. of Terms Relating to Materials for Roads and Pavements (D 8 - 33)

Committee D-8 on Bituminous Waterproofing and Roofing Materials:

Report presented, in the absence of the chairman, by W. F. Fair, Jr., and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Asphalt Roofing Surfaced with Powdered Talc or Mica (D 224 - 44 T)

Spec. for Asphalt Roofing Surfaced with Coarse Mineral Granules (D 249 - 45 T)

Sampling Bituminous Materials (D 140 - 41 T), jointly with Committee D-4 on Road and Paving Materials

Adopted as Standard:

Spec. for Asphalt Shingles Surfaced with Coarse Mineral Granules (D 225 - 44 T), as revised

Adopted as Standard, Revisions in:

Spec. for Wide Selvage Asphalt Roofing Surfaced with Coarse Mineral Granules (D 371 - 44)

Spec. for Asphalt Siding Surfaced with Coarse Mineral Granules (D 699 - 44)

Committee D-18 on Soils for Engineering Purposes:

Report, not preprinted, presented from manuscript, in the absence of the chairman, by K. B. Woods.

Papers:

The following papers were presented:

Ten Years' Outdoor Exposure of Filled Asphalt Coatings on Saturated Felts[22]—G. L. Oliensis, presented by the author.

Development of a Method of Test for Soil Bituminous Materials—E. O. Rhodes and P. F. Phelan, presented from manuscript by W. F. Fair, Jr.

Laboratory Investigation of Anti-Stripping Admixtures Used for Promoting Wetting Power and Adhesion Between Bitumens and Aggregates—J. C. Sprague, presented by Burton Bell.

There being no further business, the Forty-ninth Annual Meeting adjourned *sine die.*

August, 1946.

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[22] ASTM BULLETIN, No. 144, January, 1947.

THE CHALLENGE OF NATIONAL AND INTERNATIONAL AFFAIRS TO THE ENGINEER

ANNUAL ADDRESS BY THE PRESIDENT

J. R. TOWNSEND[1]

June 26, 1946

The time-honored method of determining future trends is to assume that things will keep to their courses until a serious disruption causes a change in direction. By going backward a few years, it is seen that our people are looking more and more to government for the management of affairs. Recent events reveal that this may become a more and more entrenched habit of our people. All of us are mindful of the tremendous technical advances that have taken place. It now behooves us to take stock and determine if the politician and the legislator alone can be depended upon to handle the problems that arise in the path left by scientific advances. Certainly, they must be assisted by technologists to a comprehension of these new forces. Neither the engineer nor the politician alone can be expected to solve these problems. I wish to plead therefore for a more active participation of the engineer in national and international affairs.

It is clear that the rôle of the politician is to get people to do things. They collect and add direction to public will. They espouse and sharply define issues and direct public affairs.

The time-honored position of the engineer or technologist is to do the staff work. The politician looks to him for information but distrusts his advice on human affairs. The physical laws with which the engineer is most familiar are constant. The politician regards the engineer as living in an ivory tower and feels that the engineer's standards are too rigid. The rules that are used in human affairs must be elastic and adjustable to the circumstance.

The engineer, on the other hand, frequently has contempt for the politician. This contempt leads to distrust and avoidance. These, of course, are symptoms of lack of comprehension and appreciation of what the politician can do in the practical sense of getting people to work.

Out of this emerges the duty of every engineer to make his contribution to political thought. The A.S.T.M., because of the nature of its work, is particularly fitted to spearhead and show leadership in its contacts with the Government on technical matters. The engineer's most direct contact with political thought is through his client or his management and his membership in technical societies. He should think of management problems, public relations, developments in political and physical science and use every opportunity to apply his brains to the management of human affairs at his own level.

I have never pretended to be anything but an engineer. It is continually intriguing to me to solve engineering problems, and I have been stimulated by the human experience of working in a constructive and cooperative way with many engineers. There is a special significance to this. The training of the engineer should be in line with what Thomas Huxley had to say of education, namely, "the instruction of the intellect in the laws of nature under which name I include not merely things and their forces, but men and their ways." Now, the engineer can measure things and their forces; can define and describe stuff; but only much experience and association with many human beings will teach of man and his ways.

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[1] Materials Engineer, Bell Telephone Laboratories, New York, N. Y.

But engineering is not always looked upon too favorably by the intelligentsia of science. To them it seems a debased art, fortuitous at best, based on compromise and not always upon rigorous and fundamental thinking. To the man on the street, engineering is taken for granted. This impression is nearly always created by *good* engineering, since the best engineering solution is a simple one and on the face of it this looks easy. An engineer is thus seen to be a simple fellow, dealing with practical affairs with what means he has at hand, and satisfied with an agreeable solution.

There is more here than first meets the eye. The present situation in the world presents a soul-searching problem. We are overcome with *fear* and its partner, *distrust.* We feel insecure socially, politically, and economically. Each day there is revealed in the press new and more fearsome weapons. Can we trust the present leaders of men with such inexorable forces? In the field of commerce our economy is threatened by pressure groups, led by dynamic but selfish men. Despite arresting protests, these leaders tend to deprive us of choice. No opportunity is presented for consultation and negotiation, but instead we have the unpleasant spectacle of unrestrained human nature. Yet, the World's work requires that men be organized, marshalled, directed, and led if our civilization is to succeed.

I am not suggesting here that these problems be turned over to the engineer. We engineers are a modest lot. Experience has chastened and taught us through our many mistakes that trouble is to be foreseen and that human reform is slow at best. But material civilization is moving on. There are many new things to be assessed and weighed. No longer can we solve our engineering problems with Newtonian mechanics and elementary chemistry. On the surface it seems fashionable for our young engineers to be concerned with electronics, plastics, supersonics, atomic physics, and the like. An engineer cannot be a master of all of these fields of activity. However, he must acquire knowledge of their possibilities and limitations and understand how they may be adapted and applied to human use.

Please do not misunderstand me. It is not necessary for an engineer to be skilled in every subject under the sun. Capacity will automatically limit this for the individual. I do not feel either that the engineer should follow all of the sociological and economic fads of the present. A well-balanced ration of thinking, as applied to human needs, will take care of this.

The way of the legislator, and public servant, has not been made any smoother by recent events either. It has long been the tradition in America that social and economic reform can be brought about by political reform. This has involved the time-honored process of voting out the "ins." Alas, this will provide little in the solution of our problems. Knowledge of political philosophy alone and its practical application, while an important factor in the field of affairs, is not sufficient and cannot solve such problems as now beset all of us. Science and technology have provided us with potent things that must be directed for the benefit of *all human* kind. We thus have a triumvirate: *Government*, *business*, and a third party who is a master of technology. I submit that this third party should be the engineer.

Throughout history, the most potent force for binding men together has been fear. The striving for security has kept families, tribes, and nations together. We, in America, have populated a new continent, possessing good climate, rich lands, and great mineral wealth. The exploitation of this natural wealth, under our private enterprise system, has brought security and prosperity. We engineers have had good clients. America has more engineers than any other nation.

It was my privilege last spring to visit defeated Germany and to investigate at first hand its scientific and engineering methods. My approach to this problem was objective. I prepared myself for the trip by setting forth in my mind the principal problems of engineering materials in the field of communications which was to be my specialty. It would thus be possible to recognize and compare their methods of solution of these problems with our own. This proved in experience to be a fruitful approach and much of importance was revealed. But the over-all sociological picture presented by Germany was overpowering and especially significant in our present discussion.

The German system required a long apprenticeship and hence their ordinary workmen were highly skilled. This had the effect of reducing the need for supervision, detailed drawings, process specifications, and engineering. There were many technically trained people in Germany, and since there were few head positions to aspire to, there was much competition in the lower technical ranks and those that won out were very competent people indeed. Whereas this system produced much individually fine work, their production methods had much less output than ours. They had little knowledge of high-speed production machinery and hence they had to have recourse to more and more hand labor. This led to the drafting and use of slave labor. Every plant I visited had from ten to eighty per cent such slave labor. In spite of the many new devices and the excellent utilization of science, Germany was defeated. This defeat was due more to logistics, to the overwhelming weight of our arms, and thus to our industrial production and productive capacity than to the use of new weapons.

There is here a most interesting paradox which caused me much speculation. They had excellent science, an ordered society, autocratic rule, scientific direction; yet they failed. The answer lies in this. There was a profound difference between the German and the American ideology. The Germans had authority, obedience, efficiency, and a ruthless scientific logic. There was no place for sentiment or humanity in their philosophy. The American philosophy, while subject to close Governmental control and discipline, yet permitted self-determination. We harmonized the principle of self-determination with efficiency by invoking the team spirit where every man working together can contribute his best. The Germans by their use of authority, slave labor, and utter disregard for human values, completely ignored psychological for material values. Our system provided the hope for human development. Theirs contained no hope but for the elite. It should be clear that our system provided freedom of choice, thus stimulating achievement. Human values were not subordinated to mechanical efficiency. Herein is a profound lesson.

It is folly to suggest that only the technical man should manage or direct public affairs. Human affairs must be settled by compromise and adjustment. The answer is never a rigorous one except in the broad principles based on the proper human selection of values. The rigorous method of the scientist will not do. On the other hand, the legislator does not understand or appreciate the nature of modern industrial processes or developments. He needs the help of the technologist to reveal the facts so that he may assess the human values.

Here, then, is the problem in simple terms. The Nazi ideology had used science wrongly. We must assess the facts of science and set the proper human values. I now ask you, why not have the engineer complement to a much greater extent the thinking of the legislator?

In a significant article by Morris L. Cooke in the September, 1945, issue of *Mechanical Engineering*, it is demonstrated that the engineer, while being the best equipped to adopt such a rôle, is in fact less concerned with public affairs than almost any other class of individual. The situation thus portrayed is shocking indeed.

Among all of the technical societies, it seems to me that A.S.T.M. is the best training ground for the engineer to acquire the experience needed in compromise and evaluation of the facts of science in human affairs. The work of our Society deals in devising and adopting methods of test. Only by such tests can we judge the value of an improvement or compare two or more products for a specific use. These tests are adopted only after very careful study, using the results of a number of investigators. Modifications are made and compromises are permitted so that all concerned may find it convenient to carry out the tests. These tests are then applied to a product, and tolerable limits are set by conference between producer and consumer and a new standard is born. Commercial selfish interests are in the background during this process. The steps are clear: first, recognition of a need; then investigation of facts, compromise and adoption. Experience provides limits and these are adjusted to form a new standard. All of the work is voluntary. There is no coercion.

During the war, a vast number of standards, emergency standards, and emergency alternate provisions were devised and promulgated. Many of our specifications and tests were used without change by the Government. Some were developed at the specific request of the Government to meet some special need. Many of our members advised Governmental bureaus on a host of subjects. This work was in the best American tradition—cooperative, informative, constructive, and freely given.

In the philosophy of the future work of A.S.T.M., cooperation with Government departments looms large as well as with the scientific and engineering work of the other technical societies. Our Society is taking an active interest in this work and will be prepared to supply engineering tests as may be needed. It is likely that legislation will be necessary to bring the advantages of standardization to the ultimate consumer. Members of our Society should be prepared to assume the responsibility of the third member of this new triumvirate of Government, business, and technology.

As individual engineers and as members of A.S.T.M. it is our duty to ourselves as Americans, and to our profession as engineers, to take a more active part in public affairs. We can all help no matter where we are located or how situated. We can aid our fellow citizens and our legislators to assess the facts of modern technology and to assist in the comprehension of true human values so that our fellow citizens may, in the exercise of the privilege of selecting what they think best, have the opportunity to use the best intelligence possible.

In conclusion, may I offer one more quotation from Thomas Huxley:

"And, if the evils which are inseparable from the good of political liberty are to be checked, if the perpetual oscillation of nations between anarchy and despotism is to be replaced by the steady march of self-restraining freedom; it will be because men will gradually bring themselves to deal with political, as they now deal with scientifical questions; to be as ashamed of undue haste and partisan prejudice in the one case as in the other; and to believe that the machinery of society is at least as delicate as that of a spinning-jenny, and as little likely to be improved by the meddling of those who have not taken the trouble to master the principles of its action."

ANNUAL REPORT OF THE EXECUTIVE COMMITTEE*

The Executive Committee reports upon Society activities in a year that has seen the termination of actual warfare, but not the establishment of peace, nor hardly more than an approach to a condition of economic and social stability. Most of the year since the last annual meeting has comprised a post-war period in which actual war emergencies were ended so far as material production is concerned; and although there are still critical shortages in certain materials, it has been considered desirable to "reconvert" Society activities as quickly as possible to a peace-time basis. An important step in this direction is the administrative and staff reorganization, discussed later, which it is expected will contribute in an important way to the "streamlining" of organization and procedure to aid our technical activities. This is especially true of the functioning of the administrative committees and the staff men who will maintain contact with the technical committees.

The value to the members of the opportunity of meeting for presentation and discussion of data on tests of materials, for the formulation of standards, and for renewal of personal contacts and exchange of experiences in the materials field was forcibly brought home to us in 1945 when this opportunity was denied by war-imposed restrictions on travel and hotel facilities. Accordingly, the large gathering of A.S.T.M. members in Pittsburgh last February for the Spring Meeting and Committee Week—the first since the annual meeting in June, 1944—was a most welcome event; and this year's annual meeting, for which one of the finest technical programs in the Society's history has been developed, together with a most promising exhibit of testing apparatus and equipment, may well bring to Buffalo the largest attendance we have yet had. Certainly interest and anticipation in the opportunity for the first such meeting in two years is high throughout the membership—a most healthy sign of the fundamental vigor and strength of our Society. This opportunity to resume the functioning of A.S.T.M. through its annual meeting will contribute its own important part to the acceleration of our activities previously mentioned.

In many ways the committees of the Society have converted quickly from a war-time to a peace-time basis, as will be evident from the reports for this annual meeting. Research projects dropped during the war are being resumed, and new work based on technologic advances from war researches is being started. A more normal schedule of meetings has been possible for most committees; improved transportation conditions have contributed to better attendance, and at committee meetings this year there has been in evidence a keen interest in seeing that progress is made along all lines.

A reading of this report and of the appended reports of the administrative committees will reveal much progress in the expansion of technical activities, evidenced by the organization of new technical committees and by the plans of the administrative committees dealing with simulated service testing and

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[*] Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

ultimate consumer goods. District activities have not been neglected, and this important side of the Society's functions will be greatly developed in the coming years.

The acquiring of permanent headquarters in a building owned by the Society marks an important milestone in our history—made all the more significant by the manner of its establishment through the generous gifts of the members themselves. A more convincing demonstration of the value of the Society to its members and the regard in which it is held could hardly be experienced, and the desire of the members to see the headquarters established in a setting commensurate with the position of the Society in the science and industry of our country has been an inspiration to the Executive Committee.

The Executive Committee has devoted much time to the implementation of policies announced a year ago, and the more significant accomplishments are presented in subsequent sections of this report.

Much thought is also being put on planning for the foreseeable developments of the future, with particular consideration to the financial aspects of such planning. A reading of the later section of this report devoted to 1946 finances will show how the financing of our immediate expansion is planned, that is, by drawing upon surplus that has been set aside in recent years for just such a purpose. As noted there, it can be expected from all past experience that new technical activities will in due time pay their own way—indeed, this is the only practicable way in which new work can be undertaken. Expansion beyond that immediately in hand can readily be foreseen for several years ahead, and this too should be supported by a higher level of income resulting from increased membership and publications developed from the new activities. But, coincident with this technical expansion—and the concomitant expansions of staff—we are already entering a period of higher costs, both for services (wages and salaries, our own and those of others affecting services to us) and goods (publications, supplies, etc.). In the one item of publications alone, the costs for 1946 in various categories of printing have increased from 10 to 20 per cent. The adjustment of wage and salary scales to present trends, together with staff expansion, has increased the salary budget for 1946 by approximately 20 per cent over 1945. Other expenses are growing in proportion. These considerations point to the necessity of adequate financing of Society operations; the increased costs must be met from increased income, of which the three principal sources are membership dues; publication sales, and advertising. Careful attention is being given to the pricing of publications, especially for subscription or sale to non-members, commensurate with their growing intrinsic values and the costs of printing them. Advertising rates for our publications are being examined. The Executive Committee is also studying the adequacy of the present membership dues in relation to the publications and other services that the members receive for their dues, a problem that is intimately associated with the methods of publishing the papers, reports and standards of the Society and with determining "what" and "how much" publications a member should receive for the dues. The problem also suggests the need for a far broader membership base than we have at present, despite the steady growth of the past five years.

The importance of these matters to the well-being and sound development of the Society is obvious, and they will receive the close attention of the incoming Board of Directors as it takes up its duties after the annual meeting.

Final Report of Study Committee:

The work of the Special Study Committee has been described in the two preceding annual reports, where references were made to the recommendations presented in three interim reports to the Executive Committee. Thus, the 1945 report cites policies adopted and put into practice upon the basis of Study Committee recommendations, among which are extension of work in the field of ultimate consumer goods, new work undertaken in the field of simulated service testing involving parts and assemblies, the reorganization of the administrative set-up of the Society, including headquarters staff, and changes in the publication of technical papers and discussions and of the BULLETIN.

The final report of the Study Committee was presented to the Executive Committee at the quarterly meeting in January, 1946, at which the Executive Committee considered all comments and recommendations that had not been covered in previous reports. The principal Society activities and problems thus dealt with include: Membership, meetings, promotional activities, technical committee organization and functioning, research activities, certain cooperative relations with the Federal Government, the formation of technical divisions in the Society, the activities of the Cement Reference Laboratory and possible extension of such activities in other fields, and finally suggestions for new fields of work in the Society.

Perhaps the most important and far-reaching of these dealt with the organization and functioning of the technical committees, and here the Study Committee has included many suggestions and formal recommendations. Some of these will require further study and conference with the officers of technical committees, for example, proposals for reorganization of Committee E-1 on Methods of Testing. Other recommendations have been approved and will be implemented in due time, such as: Means of increasing consumer representation and active participation in the technical committees; the importance of giving more consideration in our committee work to the significance of tests; and the importance of the Society taking vigorous initiative in the development of its research and standardization programs, in expansion of its activities, and in the continued appraisal of its potential services to industry.

In accepting the Study Committee's report and in finally discharging the committee, the Executive Committee through a formal resolution expressed its thanks and appreciation to the members of the committee for their valued services in reviewing so critically and comprehensively the Society's structure and activities and developing so many constructive recommendations.

It has been decided to distribute suitably annotated copies of the Study Committee report to the members of administrative committees and to the officers of technical and district committees, and further to publish from time to time in the BULLETIN suitable articles based upon various sections of the report.

Technical Activities:

Since VE and VJ days there has been a resurgence of technical committee work that had been slowed down in the latter months of the war by restrictions on travel and meetings imposed by the Office of Defense Transportation. The end of the war emergency and the release for general industrial applications of technological developments of the war have provided many opportunities for expansion of technical activities, which is reflected in the formation of a number of new technical committees and in the publication of papers describing researches in materials.

The Society's work in the field of research was perhaps the hardest hit of any by war restrictions, and it has therefore been particularly gratifying to note the resumption by our committees of many research projects just as quickly as they could be taken up following the end of the war. Reference to administration of research work will be found in the appended report of the Administrative Committee on Research.

Standardization activities on the other hand were continued actively in many fields during the war, and the appended report of the Administrative Committee on Standards records various developments of the past year. Current committee reports record much progress, both in new tests and specifications and in revisions to keep abreast of progress.

The Executive Committee believes it desirable that the emergency alternate provisions and emergency standards, which served such a useful purpose during the war, should be discontinued now that the emergency is over. All technical committees were therefore advised that these emergency requirements should be discontinued as such after July 1, 1946. Accordingly the technical committees having such emergency requirements under their jurisdiction have been engaged in reviewing these items to determine whether they should be dropped or incorporated in the standards. The committees will report their recommendations in these matters either to the Annual Meeting or to the Administrative Committee on Standards. The Executive Committee is prepared to consider any instances where a committee feels strongly that some exception to this general policy is necessary.

In connection with this action the Procedure for Emergency Provisions and Standards[1] has been discontinued.

The appended reports of the Administrative Committee on Simulated Service Testing and the Administrative Committee on Ultimate Consumer Goods record important developments in these two new fields of work upon which the Society has entered this past year. The organization of three new committees of the Society has already developed from the work of these two administrative committees. On the recommendation of the Administrative Committee on Simulated Service Testing the following new committees have been established: Committee E-6 on Methods of Testing Building Constructions, organized early this year under the chairmanship of L. J. Markwardt, Forest Products Laboratory; and Committee E-9 on Fatigue (which will include the present Research Committee on Fatigue of Metals), to be organized during the annual meeting under the temporary chairmanship of R. E. Peterson, Westinghouse Electric Corp. In organizing this committee, it is not intended that current work on this subject in certain committees of the Society be discontinued; on the contrary it is desired to stimulate all such work and it will be a function of the new committee to coordinate the development of fatigue tests by the technical committees.

As a development from discussions in the Administrative Committee on Ultimate Consumer Goods, steps are being taken to organize a new technical committee on Engine Antifreezes. A preliminary meeting of selected producers and consumers of these products was held early this year in Pittsburgh to explore the field and suggest a scope of activities for such a committee. On the basis of the recommendations of this group the Executive Committee has authorized the organization of the committee and has designated H. R. Wolf, General Motors Corp., as temporary chairman. The following scope has been tentatively approved:

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[1] 1945 Year Book, p. 454.

> The study of engine antifreezes. This will include terminology, identification and classification; methods of sampling and testing; interpretation and significance of tests; and the preparation of specifications.

On the recommendation of the Administrative Committee on Standards a new technical committee on Asbestos-Cement Products was organized on June 6 under the temporary chairmanship of D. E. Parsons, National Bureau of Standards. The following scope for the committee is under consideration:

> The formulation of specifications, definitions and methods of test for asbestos-cement sheet, shingles, siding, and other products.

The organization of this committee was determined upon after an extensive canvass of both producers and consumers of such products, which established clearly that asbestos cement is an important building material for which there is definite need for methods of test and specifications.

The Executive Committee has also authorized the organization of a new technical committee on Oxychloride Cement. This decision is related to the organization under the joint sponsorship of this Society and the National Bureau of Standards of a Sectional Committee on Specifications for Installation of Oxychloride Cement Flooring under the procedure of the American Standards Association. A survey of this field established the fact that there is a clear need for tests and specifications for oxychloride cement as such, not alone for use in flooring but in connection with other uses of this type of cement, such as with refractories, waterproofing and heating compounds. L. S. Wells, National Bureau of Standards, has been designated as chairman *pro tem* and selection of the personnel of the committee is now being made.

In its report a year ago[2] the Executive Committee referred briefly to war-time developments in quality control of materials and their significance to the work of the Society and announced a decision to form a separate standing committee of the Society on this subject. During the year this subject has been studied by an organizing committee appointed by the Executive Committee and consisting of H. F. Dodge, chairman, A. G. Ashcroft, G. H. Harnden, H. F. Hebley, F. T. Mavis, R. F. Passano and A. E. R. Westman. On the recommendation of this group the new committee is now being organized as Committee E-11 on Quality Control of Materials, and will function under the following statement of scope:

> The committee is organized to promote the knowledge of quality control methods and their application to specifications and methods of test. By quality control methods is meant those methods that have been developed on a statistical basis to control the quality of product through the proper relation of specification, production, and inspection as parts of a continuing process.
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> The committee is authorized to sponsor papers and discussions; to prepare reports, manuals and recommended practices; and to aid and advise the committees of the Society on the application of quality control methods in: (*a*) the collection, analysis, interpretation, and presentation of data; (*b*) preparing specifications and methods of test; (*c*) establishing specified limits in specifications, including the designation of numerical requirements; (*d*) preparing acceptance sampling plans to be used for determining conformity to specifications; and (*e*) setting up sampling plans for control of quality in manufacture.

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[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 21 (1945).

It has been considered desirable that the membership of the committee should be limited for the present to 25, with the understanding that subcommittees established to work on specific projects will draw membership from qualified members of interested technical committees of the Society. A formal meeting for organization was held on June 10 under the temporary chairmanship of H. F. Dodge, Bell Telephone Laboratories. With the organization of this committee there has been transferred to it responsibility for the work of two committees of Committee E-1 on Methods of Testing, namely, those on Interpretation and Presentation of Data, and Designation and Interpretation of Numerical Requirements.

The Executive Committee is discussing with Committee E-1 on Methods of Testing plans for the reorganization of that committee, based in part upon review by the Study Committee of the rôle in Society affairs of this important committee on testing. The committee work of the Society has grown so much in the 25 years since Committee E-1 was organized on its present basis that a reappraisal of the work of the committee and its relation to the work of other committees of the Society is needed.

Committee D-2 on Petroleum Products and Lubricants has during the year expanded its work to cover the development of specifications for diesel fuel, tractor fuel, and aviation fuel, for which purpose the committees working in these fields will be made much more widely representative of the consuming interests. The committee is also creating a new group to be responsible for work on recommended practices for procurement of lubricants. On the recommendation of the Administrative Committee on Standards, Committee D-2 has undertaken the organization of a new committee on cutting oils.

With the increasing complexity of industrial materials and constructions we are finding that scopes of some of the technical committees overlap to an increasing degree, requiring adjustments by the Executive Committee. Several such cases were taken up during the year, as follows:

The reorganization of Committee D-10 on Shipping Containers, as noted in the report a year ago, presented a problem of overlapping of jurisdiction with Committee D-6 on Paper and Paper Products. It has been decided that Committee D-10 shall have jurisdiction for all work in the Society on the development of tests and specifications for containers, which will include containers made of fiberboard, but that the development of tests and specifications for fiberboard itself shall remain in the jurisdiction of Committee D-6. Certain methods of test for containers developed by the latter committee have been transferred to the jurisdiction of Committee D-10. It is understood that the two committees will cooperate one with the other so that there is proper coordination between the tests and specifications for fiberboard on the one hand and tests and specifications for fiberboard containers on the other.

The development of Methods of Test for Ply Adhesion of Paper and Vulcanized Fiber prepared by Committee D-6 on Paper and Paper Products raised the question whether such portion of these methods as relate to vulcanized fiber should be in the jurisdiction of Committee D-9 on Electrical Insulating Materials, which has worked upon such products for a number of years. A conference of representatives of the two committees was arranged. It has been agreed that vulcanized fiber is essentially a paper. Therefore, jurisdiction with respect to mechanical tests of vulcanized fiber and vulcanized fiber products has been assigned to Committee D-6, whereas Committee D-9 will continue to have jurisdiction over all electrical tests of vulcanized fiber as well as the preparation of specifications for vulcanized fiber for electrical uses. It is understood that frequently Committee D-9 can make use of general specifications prepared by Committee D-6, incorporating such additional requirements as might be needed so far as electrical uses are concerned. In connection with this ruling the Executive Committee has asked that Committee D-6 increase the representation of producers and consumers of vulcanized fiber on that committee.

Consideration of this subject has led to a suggestion that the work of the Society in this and similar fields might be facilitated through the establishment of a coordinating committee dealing with materials falling within the scope of such committees as D-6, D-9, D-20 on Plastics, D-7 on Wood, C-16 on Thermal Insulating Materials, D-11 on Rubber and Rubber-Like Materials, and possibly D-13 on Textile Materials. This suggestion is at present under consideration.

The field of work assigned to Committee D-16 on Industrial Aromatic Hydrocarbons overlaps that assigned to Committee D-1 on Paint, Varnish, Lacquer, and Related Products in that these hydrocarbons are used in the manufacture of products within the scope of the latter committee. It has been decided in conference with the two committees that standards covering aromatics may be developed by Committee D-1 as they relate specifically to application to paint, varnish, lacquer and related products, while it will be within the jurisdiction of Committee D-16 to develop specifications of general character for all the aromatic hydrocarbons. To facilitate the adjustment of possible future questions of jurisdiction between the two committees and to harmonize any conflict in standards under the jurisdiction of each, there has been established a joint conference committee of the two committees.

Since different units for measurement of vapor transmission are currently used in different industries, for example, one system being current in the field of heating and ventilating engineering and another with respect to various paper products, there has been formed a liaison subgroup among the following committees of the Society to correlate their work on expression of vapor transmission measurements: C-16 on Thermal Insulating Materials, D-6 on Paper and Paper Products, D-1 on Paint, Varnish, Lacquer, and Related Products, D-10 on Shipping Containers, and D-20 on Plastics.

During the year it was decided by agreement between the two committees concerned that specifications for deoxidizers and hardeners for copper alloys (B 51, B 52, B 53) would be transferred from Committee B-2 on Non-Ferrous Metals and Alloys to Committee B-5 on Copper and Copper Alloys, Cast and Wrought, which will hereafter be responsible for such materials. In accordance with this decision the scope of the latter committee has been enlarged by including suitable reference to these materials. The modified scope was printed in the 1945 Year Book.

Minor changes in scopes of Committees D-13 on Textile Materials and D-16 on Industrial Aromatic Hydrocarbons were made during the year and published in the 1945 Year Book.

After appropriate consultation with the Coordinating Committee on Non-Ferrous Metals and Alloys and with certain of the ferrous metals committees who have an interest in the subject, the scope of Committee B-1 has been enlarged to cover all types of wire for electrical conductors rather than copper and copper-alloy wires as heretofore. Accordingly the title of the committee was changed to Committee B-1 on Wires for Electrical Conductors and the scope changed to read:

> Formulation of specifications and methods of test for uninsulated wire for electrical conductors, solid, stranded and braided.

It has been decided to change the designation of Committee C-5 on Fire Tests of Materials and Construction to Committee E-5, placing it in the "E" group of committees dealing primarily with methods of testing. It is felt that this change is a logical one inasmuch as the committee's activities cover a number of different types of materials and are not confined solely to the materials in the "C" group. Furthermore, in view of the fact that several of the technical committees of the Society are interested in the determination of fire hazard properties of materials within their scope, the Executive Committee has arranged that such committees in the preparation of methods of conducting fire hazard or fire-resistance tests will confer with Committee E-5 and request advice and comment on proposed methods of flammability tests.

The war greatly stimulated the development of types of construction involving the use of two or more materials in such a way as to utilize in the composite article the special individual properties of the constituent materials—types to which the term "sandwich construction" is frequently applied. In the Society, responsibility for the development of tests and specifications for the constituent materials of these "sandwiches" is vested in several committees, typical of which are those dealing with such products as sheet metal, wood, rubber, plastics, adhesives, textiles, glass, and paper. There is need for the development and standardization of adequate tests and specifications to cover these composite materials, and the relationship between such work if undertaken by the Society and the limits of scope and jurisdiction of existing committees is one that requires thoughtful consideration. The Administrative Committee on Simulated Service Testing is definitely interested because such testing is frequently necessary for these types of construction.

As a first step in the consideration of this subject, a conference of committee representatives and of producers and consumers of these types of constructions and materials was held in Philadelphia on June 7. The findings of this conference will point the way to further development of the subject.

The Executive Committee has under consideration a proposal from M. Rea Paul, the Society's representative on the Inter-Society Color Council, that the general question of Appearance Standards be considered in the Society. It is pointed out that such requirements are scattered through various A.S.T.M. standards and that in the interest of standardization it might be well to form a technical committee in the "E" group that would be charged with the responsibility of reviewing and correlating all proposed appearance specifications. The Executive Secretary has been directed to develop this subject for further consideration of the Executive Committee.

By-laws and Charter of the Society:

The letter ballot of the Society authorized at the annual meeting in 1945 to amend the By-laws in accordance with recommendations of the Executive Committee resulted in a vote of 521 affirmative, 1 negative. Accordingly, the amended By-laws became effective on

September 15, 1945, except that the substitution of a Board of Directors for an Executive Committee was contingent upon a change in the Charter of the Society.

Pursuant to unanimous action at the 1945 Annual Meeting on a resolution offered by the Executive Committee,[3] proposing amendments to the Charter to provide for the creation of a Board of Directors and authorizing the President to present Articles of Amendment to the appropriate court of the Commonwealth of Pennsylvania, the amendments to the Charter were formally approved by the court on December 10, 1945. Accordingly, under the terms of Article XI of the By-laws[4] the new Board of Directors will come formally into existence at the close of the annual meeting in 1946. The present ten Members of Executive Committee will under the terms of the By-laws have one year added to their present terms and will serve thereafter as Directors, and the Society will elect five new Directors for a term of three years beginning at the close of the annual meeting.

Administrative Organization:

During the year the Executive Committee has put into operation the plan for establishment of administrative committees recommended by the Study Committee and described in the annual report for 1945.[5] There are at present six such committees, to which are assigned certain administrative duties and responsibilities, including the important function of serving in an advisory capacity to the Executive Committee (after this annual meeting, to the Board of Directors). These committees are:

Administrative Committee on Standards (formerly Committee E-10);
Administrative Committee on Research (formerly Committee E-9);
Administrative Committee on Papers and Publications (formerly Committee E-6);
Administrative Committee on Ultimate Consumer Goods;
Administrative Committee on Simulated Service Testing; and
Administrative Committee on District Activities.

The personnels and scopes of these committees are given in the 1945 Year Book, pp. 5–6. The first three are of long standing. The last three were organized in 1945; the reasons for forming these committees and the general fields of work assigned to them were discussed fully in last year's report.

These six committees now report directly to the Executive Committee (later to the Board of Directors). Their work is of such importance that their reports are appended to this report as a matter of record of important happenings and statistics in the Society and to give expression to discussions of policy and procedure that have important relations to the life of the Society. References to these reports are made in appropriate sections of this report.

It has been decided to increase the membership of the Administrative Committee on Research from five to six members, and to change the terms of office from five to three years so arranged that the term of two members will expire each year. This change will be put into effect with this annual meeting.

Committee E-5 on Standing Committees, which consisted of the chairmen of all standing committees, has been disbanded with the consent of its members. It had become so large as to be cumbersome and unwieldy in operation. Its only function had been to share with the Executive Committee the responsibility for the Regulations

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[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 35 (1945).

[4] 1945 Year Book, p. 438.

[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 45, pp. 28 and 30 (1945).

Governing Standing Committees, which is now vested in the Executive Committee under the following conditions, which are published with the Regulations in the Year Book:

> The responsibility for the Regulations Governing Technical Committees is vested in the Executive Committee. Changes in the Regulations may be initiated either by the Executive Committee or by the chairman of any technical committee. All proposed changes shall be referred to the chairmen of all technical committees for advice and consultation before final action by the Executive Committee. Changes in the Regulations adopted by the Executive Committee shall be announced to the members and shall become effective as of dates determined by the Executive Committee.

Organization of Headquarters Staff:

In October, 1945, the Executive Committee gave effect to provisions of the amended By-laws by electing C. L. Warwick as Executive Secretary, R. E. Hess as Assistant Executive Secretary, J. K. Rittenhouse as Treasurer, and R. J. Painter as Assistant Secretary. The Executive Committee also set up the five departments of the headquarters staff as explained in the report a year ago.[6] The Technical Activities Department is headed by Mr. Hess as Technical Secretary; the Editorial Department is headed also by Mr. Hess in the capacity of Editor. The Field and Secretarial Department is headed by the Assistant Secretary; the Finance Department is headed by the Treasurer. The Office Management Department is headed by an Office Manager, to which post James H. Wolfe, Jr. was recently appointed.

In the Technical Activities Department there are serving under the Technical Secretary two assistant technical secretaries, P. J. Smith and C. S. Cole, with LeRoy C. Gilbert as technical assistant, the latter having joined the staff on April 1, 1946. In the Editorial Department there are serving under the Editor, the Standards Editor in the person of Mr. Smith; the Assistant Editor, G. A. Wilson; and two technical assistants, M. D. Huber and J. W. Caum, the latter having joined the staff on December 11, 1945.

The appointment of Mr. Gilbert provides another staff engineer who will work directly with the committees of the Society dealing with the construction materials (C and certain D group committees) and is a further step in the implementation of the policy to expand the staff work on technical activities.

The growth of the general activities of the Society, especially those concerned with membership, publication sales, editorial and publication activities, and district activities, will necessitate expansion of stenographic and clerical staff, to which the Executive Secretary is giving particular attention.

New Society Headquarters:

The Executive Committee has kept the members informed through the A.S.T.M. BULLETIN of the steps that have been taken since the last annual meeting in the purchase of the property at 1916 Race St., Philadelphia, for the new headquarters, and of the progress of the alterations and additions to the property to fit it to the requirements of headquarters occupancy. This report will therefore be confined to a brief résumé and a statement of present status.

The Society acquired title to the property on August 1, 1945, and by the middle of October the building had been vacated of all tenants. Plans and specifications for the alterations and additions were prepared by Sylvester L. Smith, who was retained as the Society's architect; and the general contract was let on November 14, 1945, to William D. Baker Co. The Headquarters Committee that had negotiated the purchase was continued as a Committee on Building with some expansion of membership, and this committee has been in charge of the building project for the Society. The personnel of the committee comprises Messrs. J. F. Vogdes, Jr., chairman, E. J. Albert, T. A. Boyd, A. W. Carpenter, G. H. Clamer, P. H. Dike, Alexander Foster, Jr., and J. R. Townsend.

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[6] *Proceedings*, Am. Soc. Testing Mats., Vol. 45, pp. 30–31 (1945).

Progress on the work has been disappointingly slow, partly due to local labor strikes and to general strikes and shortages of materials, although in this latter respect we were fortunate in securing an adequate supply of basic construction materials, such as brick, lumber and steel. Accordingly the building is still far from complete. Through the courtesy of the Atlantic Refining Co., the Society's lease on headquarters in the Atlantic Building was extended to the end of May, by which time the center and rear portions of the building throughout all floors and the basement were sufficiently completed for occupancy. The move to the new headquarters was completed on May 27. Work on the remainder of the building is now proceeding more rapidly and although it may be some months before certain construction and installation of equipment can be completed, notably air-conditioning, it is hoped that the building will be substantially ready for complete occupancy before the end of the summer.

It is planned to publish in the A.S.T.M. BULLETIN a description and plans of the building, including the arrangement of general offices, library, facilities for storage and distribution of publications, as well as facilities provided for officers, directors and members of the Society and for conferences of administrative and technical committees.

The move into the new headquarters will necessitate the purchase of much in the way of new furnishings for offices, members' and committee rooms, and the entrance lobby. All of these furnishings have been ordered.

Financial Aspects.—The entire headquarters project has been made possible by the generous contributions of the members of the Society to the Building Fund. On June 1, 1946, these contributions had reached the total of $150,118.43. It had confidently been expected that this amount would more than meet all of the costs involved in the purchase and alterations; but the great increase in building costs over the past eight months has been such that original estimates of costs are being exceeded by amounts ranging from 30 to as much as 50 per cent.

Despite changes in the original plans that were made in an effort to economize in the construction, it was evident in April that to complete construction of the headquarters building as desired would require an additional $25,000 to $30,000. The Executive Committee decided that the building should be completed as planned and as it believed the members would wish to have it, and has placed in the hands of the Finance Committee the problem of providing the needed additional monies. As a first step the Finance Committee has directed the Executive Secretary to bring the situation to the attention of the membership with an appeal for contributions to the Building Fund on the part of members who have not yet contributed. In the meantime other sources of the needed funds are being examined.

As noted in a later section of this report, all financial operations in connection with the purchase and alterations of the building are handled in a Building Fund, which is entirely separate from all other funds of the Society.

The Executive Committee gave careful consideration to the possibility of separate incorporation of the headquarters building, having principally in mind such questions as liability of the Society and its assets in case of serious casualty on the premises, as well as possible advantages in having ownership and operation of the building vested in a separate corporation. On advice of the Society's legal counsel it was decided not to undertake such separate incorporation, inasmuch as this would not exempt the Society from liability and would involve the Society in certain tax obligations, additional bookkeeping and greater expense. The Society is adequately protected by the public liability insurance policies that are being carried.

District Activities:

An important development in district activities during the year was the organization of a new District Committee covering the New England area, excluding those counties in Connecticut already a part of the New York A.S.T.M. metropolitan district. Following an informal conference with some of our leading members in the New England area and an analysis of the results of a questionnaire sent to all members in New England, it was apparent that a district organization would receive excellent support. The organization meeting was held on May 20, 1946, with President Townsend, Secretary Warwick, and the Chairman of the Administrative Committee on District Activities, C. H. Fellows, present. Further details of this district meeting will appear in the August A.S.T.M. BULLETIN. This is the eleventh district to be organized.

District Meetings. — From the accompanying table of A.S.T.M. meetings, it will be noted there were numerous sessions planned by the various districts. Unquestionably the outstanding of these affairs was the one in Detroit on October 25, 1945, with addresses by President J. R. Townsend and Dr. C. F. Kettering, where the attendance of 925 was the highest yet recorded for any A.S.T.M. session, district or national. The Pittsburgh district cooperated in connection with the Spring Meeting, and arranged the technical discussion on statistical quality control. Interesting groups of technical papers were presented in the Symposium on Effect of Low Temperatures on the Properties of Materials, arranged by the Philadelphia District in March, 1946, and the sessions on simulated service testing arranged by the Chicago District created much interest.

It will be noted from the table of meetings that many of the papers presented at district meetings have been published in the A.S.T.M. BULLETIN. The presentation of such a variety of technical information with subsequent publication of papers and addresses, is significant. These district meetings to an increasing extent are affording forums for discussion of significant aspects of the Society's work, particularly in relation to the testing and evaluation of materials. President Townsend has taken great interest in the district work, and he was the chief speaker at several of the meetings, presenting interesting addresses on Research Revolutionizes Materials and Research at Home and Abroad. His critical analyses of the methods used by the Germans and our own research and standardization activities were of interest to all of the groups which he addressed. The Executive Secretary or some other member of the Headquarters Staff has usually attended the district meetings with the President.

In line with the desire of the Executive Committee that there should be fostered whenever possible closer relations with the Pacific Coast members, the President and Executive Secretary made visits to the Southern and Northern California Districts in March. Many worth-while contacts were made and the work of the Society was stressed through addresses at the Los Angeles and San Francisco meetings.

The mere holding of district meetings would not of itself be justified were it not that they provide for members to get together, many of whom cannot get to a national meeting, and they bring to the usually large number of visitors at the sessions a much better concept of the important work the Society is doing.

The Executive Committee desires to record its appreciation for the loyal efforts and cooperation of the district committee officers and members. Many of them are active in other A.S.T.M. work, and the interest which they take in district work is indicative of the importance with which this is viewed by our members.

Special acknowledgment should be made of the very valuable service being rendered by the Buffalo Committee on Arrangements for this Annual Meeting. This committee, which is an augmented group based on the Western New York-Ontario District Committee, has been active in connection with many phases of the meeting, including the Annual Meeting Dinner, guest speaker, the two exhibits (Apparatus and Photographic), industrial trips, golf tournament, and entertainment. Under the guidance of T. L. Mayer, Buffalo Public Library, who is Secretary of the District, and Acting Chairman of the Committee on Arrangements due to the illness of District Chairman B. L. McCarthy, a well-integrated and efficient working group has been functioning.

Administrative Committee on District Activities.—The administration of district affairs has been delegated by the Executive Committee to the Administrative Committee on District Activities, whose brief report of current developments is appended hereto. Reference is made in that report to the review of the Charter for Districts that is in course of revision to give the districts and their governing bodies greater autonomy. A Manual of District Activities which is in preparation will be a valuable guide to district officers in conducting their work.

Other subjects that are under consideration in the Administrative Committee include a more exact determination of district areas and a study of where new districts might effectively be organized to aid in promoting the work of the Society. The Administrative Committee is also studying the general question of participation of our various districts in the activities of local technical society councils or federated engineering groups. The Cleveland, Chicago, Detroit, and Pittsburgh Districts are already affiliated with such groups; it is expected that the New York District will affiliate with a technical society group now being organized, and that in due time the New England District will affiliate with the Engineering Societies of New England.

National Meetings:

The annual meeting of the Society in 1945 was a curtailed one because of the war and the transportation crisis and consisted only of a one-session business meeting in New York at the Hotel Pennsylvania on June 27, with a registered attendance of 90 members. The 1946 Spring Meeting was held February 26–27, 1946 in Pittsburgh. This meeting, held during A.S.T.M. Committee Week (throughout the week of February 25) comprised three sessions: one a discussion on Statistical Quality Control and Its Application to Specification Requirements, and two sessions comprising a Symposium on Atmospheric

A.S.T.M. MEETINGS JUNE, 1945—MAY, 1946, INCLUSIVE

Meeting	Place	Date	Notes
Annual Meeting	New York, N. Y. Hotel Pennsylvania	June 27, 1945	Abbreviated Business Meeting (See New York District Meeting noted below)
Spring Meeting	Pittsburgh, Pa. Hotel William Penn	February 26, 27, 1946	Discussion on Statistical Quality Control[a] (two papers) (Attendance about 350) Symposium on Atmospheric Exposure Tests on Non-Ferrous Metals[b] (two sessions) (About 150 in attendance at each session)
A.S.T.M. Committee Week	Pittsburgh, Pa. Hotel William Penn	February 25–March 1, 1946	200 Committee Meetings. (1000 in attendance)
District or Local (sponsored by Groups in Cities Indicated)	New York, N. Y. Hotel Pennsylvania	June 27, 1945	(Held in conjunction with the 1945 Annual Meeting) Presidential Address,[c] Preparation for the Future, P. H. Bates; Technical Feature, Preparing the Way for Victory with the Seabees,[c] Rear Admiral Lewis B. Combs. (350 attendance)
	Chicago, Ill. Engineering Building	October 24, 1945	From Research—New Facts—New Materials—New Products—President, J. R. Townsend,[d] T. A. Boyd,[e] Vice-President; Award of Charles B. Dudley Medal to W. N. Findley. (225 attendance)
	Detroit, Mich. Rackham Memorial Building	October 25, 1945	Research Revolutionizes Materials, C. F. Kettering,[f] J. R. Townsend.[d] (900 attendance)
	Pittsburgh, Pa. Mellon Institute Auditorium	November 29, 1945	Research at Home and Abroad Research Revolutionizes Materials,[d] by J. R. Townsend; Tracking Down Research in Wartime Germany, by A. A. Bates. (300 attendance)
	Cleveland, Ohio Cleveland Engineering Society Auditorium	December 4, 1945	Materials . . . Research and Production Research Revolutionizes Materials,[d] by J. R. Townsend; Wartime Materials Developments and the Postwar World,[g] by J. C. De Haven. (100 attendance)
	New York, N. Y. Engineering Societies Bldg.	December 11, 1945	The War Pays Benefits to the Motorist, by J. Bennett Hill. Also, two special films. (200 attendance)
	Northern California (San Francisco) Engineers Club	January 29, 1946	What is New in Science and Engineering,[h] by E. S. Lee. (100 attendance)
	Philadelphia, Pa. Franklin Institute	February 14, 1946	Housing and Housing Materials,[h] Walter C. Voss; J. T. Grisdale. (200 attendance)
	Western New York-Ontario (Buffalo) Hotel Statler	March 7, 1946	Housing and Housing Materials,[h] Walter C. Voss; J. W. Kideney. (100 attendance)
	Southern California (Los Angeles)	March 19, 1946	Materials Testing Here and Abroad,[i] by President J. R. Townsend; A.S.T.M. Consumers Goods Program and Service Testing, by Executive Secretary C. L. Warwick. (150 attendance)
	Philadelphia, Pa. Franklin Institute	March 19, 1946	Symposium on Effects of Low Temperatures on Properties of Materials[b] with five papers. (Two sessions) (200 attendance)
	Chicago, Ill. Stevens Hotel	March 22, 1946	Production Conference. Panel sponsored by A.S.T.M., on Simulated Service Testing of Materials and Assemblies, with papers by Alan Freas,[k] J. O. Almen, and A. T. Chameroy.[l] (200 attendance)

A.S.T.M. MEETINGS JUNE, 1945—MAY 1946, INCLUSIVE—*Concluded*

Meeting	Place	Date	Notes
District or Local (sponsored by Groups in Cities Indicated)—*Continued*	Northern California (San Francisco) Engineers Club	March 25, 1946	Jointly with San Francisco Section A.W.S. Materials Engineering Here and Abroad,[i] by J. R. Townsend; New Developments in A.S.T.M., by C. L. Warwick. (100 attendance)
	New York, N. Y. Engineering Societies Bldg.	May 2, 1946	Interesting Developments and Observations in Foreign Countries[m] (three speakers)—J. R. Townsend, D. E. Douty, W. L. White. (135 attendance)
	New England (Boston, Mass.) Engineers Club	May 20, 1946	Organization Meeting of New England District, with informal talks: The A.S.T.M. of the Future, by Executive Secretary C. L. Warwick; What is Industry's Stake in the A.S.T.M., by President J. R. Townsend. (60 attendance)

[a] Published in ASTM BULLETIN, No. 139, March, 1946; also reprinted.
[b] Issued as a separate publication, November, 1946.
[c] Published in ASTM BULLETIN, No. 135, August, 1945.
[d] Mr. Townsend's paper was published in ASTM BULLETIN, No. 137, December, 1945.
[e] Mr. Boyd's paper was published in ASTM BULLETIN, No. 138, January, 1946.
[f] Dr. Kettering's address was published in ASTM BULLETIN, No. 137, December, 1945.
[g] Published in ASTM BULLETIN, No. 138, January, 1946.
[h] Abstract published in ASTM BULLETIN, No. 139, March, 1946.
[i] Published in ASTM BULLETIN, No. 140, May, 1946.
[k] Published in ASTM BULLETIN, No. 141, August, 1946.
[l] Mr. Chameroy's paper, Laboratory Testing of Consumer Goods, published in ASTM BULLETIN, No. 140, May, 1946.
[m] Abstract published in ASTM BULLETIN, No. 140, May, 1946.

Exposure Tests of Non-Ferrous Metals. The two interesting papers on quality control have been published in the March BULLETIN, and reprints are being prepared. The corrosion symposium is to be issued as a separate publication. The active interest in both subjects was evidenced by the excellent attendance.

A.S.T.M. Committee Week in 1946 was a very intensive period with large numbers of meetings running simultaneously morning, afternoon, and evening practically all five days of the week. The renewed activities of our committees and the necessity of a great number of meetings when the Society convenes at its spring and annual meetings create numerous problems in connection with sleeping room accommodations, meeting rooms, and the scheduling of meetings to minimize conflicts—problems that will not be lessened when the newly organized committees of the Society begin their active work.

1947 Meetings.—The Executive Committee has accepted the invitation of the Philadelphia District Committee to hold the 1947 Spring Meeting in Philadelphia (the tentative date selected is the week of February 24) with an interesting symposium on paint and paint materials in prospect as the technical feature.

There is still uncertainty concerning the location of the annual meeting next year. It had been hoped to go back again to Atlantic City where so many excellent meetings have been held, but whether suitable acommodations can be obtained must be studied further. The likelihood as this report is prepared is that the meeting will be at Chalfont-Haddon Hall during the last week in June, namely June 23–27, 1947.

Pacific Coast Meetings.—When the President and Executive Secretary were on the Pacific Coast in March, there was discussion on having the Northern California District participate in some appropriate way in the Western Metals Congress in San Francisco scheduled for March, 1947; the Pacific Coast members were also interested in having a national meeting of the Society on the Pacific Coast in 1948. The Executive Committee has approved the first proposal and the Meetings Committee has been asked to consider the possibility of a 1948 national meeting on the Coast.

Publications:

The year 1945 has presented no unusual publication problems, except for difficulties more or less common to all lines of production that have been experienced in bringing publications out on time. As will be seen from lists of 1945 publications included in the appended report of the Administrative Committee on Papers and Publications, the regular publications for the year were about normal in volume for a year in which the Book of Standards Supplements are printed. It will also be seen that there were somewhat fewer compilations of standards and special publications issued than in the preceding year. On the other hand the ASTM BULLETIN continues to grow both in size and average edition, which is a natural result of the policy announced a year ago of making greater use of the BULLETIN for the publication of technical papers.

The plan of receiving offers of papers at any time throughout the year, together with immediate issue of preprints either in the 6 by 9-in. size or in the ASTM BULLETIN, has been in operation now for over a year and will be more effectively used as we gain experience with it and as prospective authors begin to appreciate that papers may be offered to the Society for publication at any time throughout the year.

The availability of the BULLETIN for publication of such papers lends greater emphasis than ever to the proposals for changing over the BULLETIN to a monthly technical journal. Such a change-over involves many important considerations and the Executive Committee has charged the Administrative Committee on Papers and Publications with the responsibility of making a complete study of and a report upon this subject not later than June, 1948.

In the meantime the change in format of the BULLETIN has made possible the inclusion in the bound volume of *Proceedings* of any technical paper published in the BULLETIN, and the 1945 *Proceedings* for the first time contain several such papers selected by the Papers and Publications Committee.

Suitable three-post expansion type binders in two sizes are now available for sale to members who wish to maintain their own selection of A.S.T.M. Standards, for which purpose all reprints of standards are now punched to fit standard three-ring binders.

Due to pressure of other more urgent publication matters, the formal institution of the Special Technical Publication Series referred to in the annual report last year has been delayed. A listing of all publications of the Society since its organization has been compiled, from which it will be necessary to select those significant technical publications that justify a listing in this series. It is planned to complete this work during the coming year and to publish the titles in the series; technical publications thereafter will be assigned a series number. The index in each annual volume of *Proceedings* now includes reference to all technical publications issued during the year.

With the publication of the 1945 Year Book there was resumed the practice of including a geographical distribution of the membership of the Society, which it is believed will be a convenience to many members and especially to the officers of the various districts of the Society.

Two important publication problems face the Society during 1946. One of these is the necessity of publishing a new edition of the Book of Standards in view of the great demand for the 1944 edition. The other is the problem of publishing the exceptionally large volume of technical papers that has been offered for the 1946 annual meeting. There has never been in the history of the Society a greater wealth of technical data offered for publication,—a situation that has come about through the release of hitherto confidential or restricted data developed through war researches. The appended report of the Administrative Committee on Papers and Publications touches further on these two important problems.

Membership:

On June 1, 1946, the membership of the Society totaled 5993, the highest figure yet reached, compared with a total of 5649 on June 1 one year ago. Statistics showing gains and losses in various classes of membership are shown in the following table:

Class of Membership	Membership		Losses				Additions		Total		
	June 1, 1945	June 1, 1946	Resignations	Dropped	Death	Transfer	Transfer	Election	Loss	Gain	Increase
Honorary	9	9	...	...	..	...	...	...	...	...	...
Perpetuity	9	10	...	...	..	...	1	...	...	1	1
Sustaining	192	194	...	...	..	...	1	1	...	2	2
Company, Firm, etc.	1466	1581	52	8		5	6	174	65	180	115
Individual, etc.	3854	4089	157	47	29	7	21	454	240	475	235
Junior	119	110	10	7		17	...	25	34	25	9[a]
Total	5649	5993	219	62	29	29	29	654	339	683	344
Student	149	138	12	87	1	15	...	104	115	104	11[a]

[a] Loss.

The net gain for the year of 344 compares with 392 for the preceding year. Elections to membership were the highest in the history of the Society and numbered 654, exceeding those for the previous year by 51; losses from resignations and delinquencies were 281 compared to 178 for the preceding year. Junior Members numbered 110 and Student Members 138.

There have been two new sustaining members as follows, bringing the total to 194:

The Ohio Steel Foundry Co.
Universal Oil Products Co.

The Executive Committee has elected to honorary membership in the Society the following six members: W. H. Fulweiler, Consulting Chemical Engineer, Philadelphia; J. O. Leech, (retired), Metallurgical Division, Carnegie-Illinois Steel Co., Cambridge, Mass.; J. S. Miller, Consultant on Asphalt Technology, Rahway, N. J.; C. S. Reeve, Technical Advisor, The Barrett Division, Allied Chemical and Dye Corp., New York City; Jesse J. Shuman, Inspecting Engineer, Jones & Laughlin Steel Corp., Pittsburgh; and Frank N. Speller, Metallurgical Consultant, Pittsburgh. They will be presented to the Society for formal award of honorary membership during this Annual Meeting.

In 1938 the Executive Committee established the plan of awarding at annual meetings certificates commemorating forty years of continuous membership in the Society. Eighty-one such certificates have thus far been awarded and this year the following 19 companies and individuals have qualified:

Duff A. Abrams
American Bureau of Shipping
American Steel Foundries
Anaconda Copper Mining Co.
Cornell University Library
Oliver C. Cromwell
Charles E. Fuller
Addison F. Holmes
The Iron Age
Harry McCormack
Norfolk and Western Railway Co.
Pittsburgh Plate Glass Co.
  Paint and Varnish Division
Ernest John Russell
Earl B. Smith
Southern Pacific Co.
Richard S. Suydam
United States Testing Co., Inc.
University of Kansas Library
University of Melbourne Library

Deaths:

The Society has lost the following 29 members through death:

	Date of Membership
Bingham, Eugene C.	1917
Blest, M. C.	1924
Doran, Albert B.	1941
Emery, A. H.	1926
Falk, Myron S.	1905
Flowers, Alan E.	1914
Fowle, Frank F.	1924
Gardner, Guy O.	1930
Geisenberger, L. H.	1942
Harding, R. L.	1945
Hardy, Charles	1943
Harper, Robert B.	1915
Kathju, Kam N.	1939
Kline, C. Benton	1945
Longstreth, Edward T.	1929
Lumpkin, L. E.	1935
MacCallum, Douglas A.	1918
Nurnberg, Charles	1924
Ogden, James C.	1924
Reifsnyder, J. W.	1921
Stoddard, J. D.	1931
Sullivan, George P.	1928
Triece, George H.	1938
Twining, F. E.	1912
Urbach, Charles	1941
Utley, S. W.	1919
Vallé, Cecilio D.	1944
Wing, Charles B.	1900
Zimmerman, J. Milton	1943

In addition to the above, the following representatives of company members of the Society passed away:

Thomas R. Akin, Lacelede Steel Co.
Kenneth E. Burgess, Columbian Carbon Co., Magnetic Pigment Division
J. F. Carle, Southern Testing Laboratories, Inc.
Charles F. Conn, Giant Portland Cement Co.
E. W. Engle, Carboloy Co., Inc.
I. T. Faucett, General Cable Corp.
R. C. Harris, Toronto Department of Works
M. E. Holmes, New York State College of Ceramics
Fred C. Lang, Minnesota Highway Dept.
Carroll R. Thompson, City of Philadelphia, Department of Wharves, Docks and Ferries

Many of these men over the years contributed a great deal to the advancement of the Society's work, particularly in connection with technical committee activities. Professor Bingham, an authority on the subject of rheology, was active in the work of Committee E-1 on Methods of Testing. Dr. Flowers, intimately concerned with many problems in materials, had a long record of service on Committees D-2 on Petroleum Products and Lubricants and D-9 on Electrical Insulating Materials. R. B. Harper, a national authority in the field of gaseous fuels, had been a very loyal member, serving on several technical committees, and was particularly interested in Committee D-3 on Gaseous Fuels. Mr. Gardner was an active worker on Committee C-1 on Cement, and Mr. Conn likewise was a long-time member of Committee C-1 and Vice-Chairman at the time of his death. Dr. Holmes was active in the work of Committee C-8 on Refractories, and Mr. Lang participated in the work of several committees including those concerned with road and paving materials and soils.

Some Membership Comments. — The Society membership is subject somewhat to the same vicissitudes which affect our national industrial life. When industry's wheels are turning at high speed there is, of course intensified interest in A.S.T.M. work, since it ties in so directly with the materials that industry produces and uses. Consequently, during the war period there was a very notable increase in the number of new members, and fewer resignations and other changes. While membership in the Society is one of the most valuable affiliations that one concerned with materials can have, nevertheless the return to peace might have had some very noticeable adverse effects on our membership. It is significant, therefore, that we can present an excellent report on membership as shown in the statistics given above, even though the resignations were higher than for the past few years.

The international influence of the Society is evident by the large number of new members from abroad, particularly the Scandinavian countries, and the renewing of many former memberships temporarily suspended during the war. We are pleased to have many of these former members back with us, and we bespeak for the entire Society, our sincere desire that the new members from abroad and our older members returning, will benefit from affiliation with the Society, and the Society from their interest, for many years of peace.

The relationship of the number of company members compared with individual memberships merits some comment because of the effect on our technical committee structure and the Society's work in general. One of the distinct advantages of a company membership, of which there are about 1775, is the privilege of designating different technically qualified individuals to serve on the committee to which the company may be elected. For the past few years there has been a very notable increase in the number of members serving on our committees who have not been personnally affiliated with the Society or serving as the official Society representative of a company member. The Society has appreciated the loyal efforts of these men and it is hoped that as time goes on many may become individual members of A.S.T.M. In response to a recent communication addressed to such persons by the President and Executive Secretary, a number have become members. The value of the Society's publications is increasing and there are distinct advantages in holding membership. Service on the Administrative Committees and the honor of election to Society office is conditioned on affiliation with the Society.

There has been intensive consideration by the Committee on Membership, headed by W. C. Hanna, of steps which might be taken, not only to increase the number of members but to make information and data published by the Society, and other services rendered, of maximum utility. In line with this general thought it is planned to include in the 1946 Year Book with the personnel of the technical committees a list of past officers of the committees. It is also planned to include a complete list of those individual and company members who have been awarded the 40-year membership certificate. To clarify the dates of membership shown in the Year Book it has been decided that when an individual membership is transferred to a company membership, the original date for the individual's affiliation will be shown as well as the new company date. Conversely when the long-time official representative of a company membership becomes a personal member, his membership will carry the date when he became the company membership representative. Many of our members cherish their long-time affiliation with the Society, and it is believed this new policy will be appreciated by many.

Finances:

Report for the Fiscal Year 1945.—The annual statement of the finances of the Society follows in the report of the auditors for the fiscal year January 1 to December 31, 1945. The report gives the balance sheet of assets and liabilities, including general funds, the newly established building fund, and special and designated funds (that is, the Research Fund, Dudley Medal and Marburg Lecture Fund, Templin Award Fund, and Committee Funds); statement of receipts and disbursements classified into "operating" (budgeted) and "non-operating"; details of special funds and the building fund; and investments of Society funds, both book and market values being given.

The Society's financial operations during 1946 were at the highest level in its history. Total operating receipts were $324,065.18. Of this amount receipts from dues and entrance fees were $126,734.75 or 39.1 per cent; receipts from sales of publications were $176,369.48 or 54.4 per cent; and receipts from miscellaneous sources, consisting principally of advertising, interest and dividends, and registration fees, totaled $20,960.95 or 6.5 per cent. Receipts from sales of publications include the large volume of sales of the 1944 Book of Standards ($72,714.31), together with receipts of $28,886.01 from compilations of standards, which continue to be an increasingly important means of publication of standards for special convenience of members and of industry generally. Sales of standards in all forms totalled almost $114,000.

Operating disbursements with all current bills paid (as detailed in the auditors' report) and including $15,000 placed in the reserve for the next Book of Standards, were $277,190.98. The difference between operating receipts and operating disbursements constitutes a favorable operating balance of $46,874.20, or about 14½ per cent of receipts, which appears as a part of the surplus as of December 31, 1945.

The auditors' report includes a statement of the financial position of the Society for the past five years. The surplus in the General Funds account, exclusive of special funds, is $165,037.47, compared to $111,606.51 a year ago. If the surplus is expressed in terms of ratio to operating disbursements, it is interesting to note that current surplus represents 60 per cent of operating disbursements, compared to 44 per cent a year ago. This is the first time for about seven years that the surplus has risen over 50 per cent of the annual costs of operating the Society. Appropriations from surplus made in the current fiscal year 1946 for various purposes are mentioned later.

The auditors' report also includes a statement of cash receipts and disbursements in the Building Fund as well as a balance sheet for the Fund as of December 31, 1945. The Building Fund has been established as an entirely separate account to receive all contributions and other income, which will be used only for capital expenditures towards the new Society headquarters. It is intended that the building will be carried on the balance sheet as an asset at full cost, including purchase price and cost of all alterations and improvements; depreciation of the building and amortization of the Fund will start on January 1, 1947, at an annual rate of depreciation which will be determined at that time. Tentatively the Finance Committee is considering a method of determining depreciation that assumes 2½ per cent annual depreciation on the value of the building proper (exclusive of land value), 10 per cent annually on the value of air-conditioning equipment, and 5 per cent annually on the value of the elevator.

Attention is called to the investment of $25,000 of the Building Fund in U. S. Treasury Bonds, which sets aside an amount more than sufficient to meet (in December, 1948) the mortgage assumed with the purchase of the property.

Investments of Society funds as of December 31, 1945, are given in the auditors' report, both book and market values being shown. The following changes during the fiscal year in the investment portfolio are noted:

GENERAL FUNDS

Bought	Cost
$2 000 Hudson Coal Co. Bonds 5s, 1962	$1 697.25
2 000 U. S. Treasury Bonds 2½s, 1967–72	2 000.00
50 000 U. S. Treasury Bonds 2s, 1952–54	50 765.63
100 shares Murray Corporation of American Cum. Pfd. 4%	5 050.06
	$59 512.94

Sold, Called or Matured	Received
$3 000 U. S. Treasury Bonds 2½s, 1965–70 (to Committee Funds)	$3 000.00
2 000 U. S. Treasury Bonds, 2½s, 1964–69 (to Committee Funds)	2 000.00
4 000 General Steel Castings Corp. Bonds 5½s, 1949 (called)	4 100.00
2 000 Pressed Steel Car Co. Bonds 5s, 1951 (called)	2 000.00
7 000 U. S. Certificates of Indebtedness 2.1.45 (matured)	7 000.00
50 shares Sperry Corporation (sold)	1 463.00
1 000 U. S. Treasury Bonds 2½s, 1967–72 (to Templin Fund)	1 000.00
	$20 563.00

BUILDING FUND

Bought	Cost
$25 000 U. S. Treasury Bonds 2½s, 1967–72	$25 390.63

RESEARCH FUND

Bought	Cost
$2 000 Hudson Coal Co. 5s, 1962	$1 697.25
30 shares Butler Bros. Cum. Pfd.	3 090.00
	$4 787.25

Sold or Accepted in Settlement	Received
$2 000 Real Estate Bonds 1315–17 Spruce Street	$830.00
50 shares Sperry Corp. (sold)	1 463.00
	$2 293.00

RICHARD L. TEMPLIN AWARD FUND

Bought	Cost
$1 000 U. S. Treasury Bonds 2½s, 1967–72 (from General Funds)	$1 000.00

COMMITTEE FUNDS

Bought	Cost
$3 000 U. S. Treasury Bonds 2½s, 1965–70 (from General Funds)	$3 000.00
2 000 U. S. Treasury Bonds 2½s, 1964–69 (from General Funds)	2 000.00
3 000 U. S. Treasury Bonds 2½s, 1967–72	3 000.00
	$8 000.00

Matured	Received
$5 000 U. S. Treasury Bonds 2¾s, 1945–47	$5 000.00

In the General Funds, profits on securities sold, called, or matured totaled $577.50, while losses totaled $47, or a net gain of $530.50. In the Research Fund there was a net loss of $1217, which includes an amount of $1170 loss in settlement of $2000 real estate bonds of the Engineers' Club of Philadelphia.

No account has been taken in the accompanying financial statement of the assets of the Society in the form of publications in stock. An inventory of technical publications as of June 1, 1946, may be summarized as follows:

		Number of Copies
Year Book		400
Proceedings		5 000
Index to Proceedings, 6 parts, total		1 286
1944 Book of A.S.T.M. Standards:		
Part I	760	
Part II	644	
Part III	541	1 945
1945 Supplements:		
Part I	1540	
Part II	791	
Part III	530	2 861
Index to A.S.T.M. Standards		7 400
X-Ray Diffraction Data Cards, sets		249
Reprints of Standards (approximate)		90 000
Compilations of Standards, Symposiums and Special Reprints (approximate)		40 000

1946 Finances.—In budgeting the Society's operations for 1946, it has been necessary to provide for the expansion of technical activities and of the headquarters staff required to keep pace with the growth of the Society's work. In addition, provision must be made for increased costs of Society operations. There are two paramount factors here: First, increases in publication costs ranging in various categories of printing from 10 to 20 per cent, together with increased costs generally of all services

REPORT OF THE AUDITORS FOR THE FISCAL YEAR, JANUARY 1 TO DECEMBER 31, 1945

Philadelphia, January 10, 1946.

MR. C. L. WARWICK, *Executive Secretary*
AMERICAN SOCIETY FOR TESTING MATERIALS
Philadelphia, Pennsylvania

Dear Sir:

We have examined the books and accounts of the American Society for Testing Materials for the year ended December 31, 1945. We did not make a detailed audit of all transactions, but made extensive tests to determine the accuracy of the records. Accounts receivable were not verified by direct correspondence with debtors. The investments owned by the Society, as detailed herein, were examined by us; all income from investments was properly accounted for.

We have prepared and submit herewith balance sheet as of December 31, 1945, statement of cash receipts and disbursements for the year ended that date, and other supporting schedules, all of which are in agreement with the records of the Society. In our opinion, these statements, with notes thereto, present fairly the financial position of the Society at December 31, 1945 and the results of its operations for the year ended that date.

Respectfully submitted,
JOHN HEINS & CO.
(Signed) I. RUSSELL BUSH
Certified Public Accountant

BALANCE SHEET AS OF DECEMBER 31, 1945
(Including Special and Designated Funds)

ASSETS

General Funds:			
Current: Cash	$79 958.60		
Less check drawn as of December 31, 1945 to cover cost of Proceedings and Supplements to Book of Standards	33 900.00		
		$46 058.60	
Investments (Market Value $166 383.37)—Cost		153 718.05	
Accounts Receivable		15 033.90	
Total Current Assets—General Funds			$214 810.55
Furniture and Fixtures (depreciated book value)			6 641.57
Total Assets General Funds			$221 452.12
Building Fund:			
Cash		$85 534.67	
Investments (Market Value $25 367.19)—Cost		25 390.63	
Accounts Receivable		466.69	
Land		30 000.00	
Building		26 503.35	
Construction Costs		860.81	
Total Building Fund			$168 756.15
Special and Designated Funds:			
Cash: A.S.T.M. Research Fund		$5 143.36	
Dudley Medal and Marburg Lecture Fund		409.43	
Richard L. Templin Award Fund		112.50	
Committee Funds		15 461.23	
			$21 126.52

Investments:

A.S.T.M. Research Fund (Market Value $42 024.12)—Cost		$38 686.24	
Dudley Medal and Marburg Lecture Fund (Market Value $6 320.63)—Cost		6 625.00	
Richard L. Templin Award Fund (Market Value $1 014.68)—Cost		1 000.00	
Committee Funds (Market Value $9 314.06)—Cost		9 000.00	
			$55 311.24
Total Special and Designated Funds			$76 437.76
Total Assets			$466 646.03

LIABILITIES

General Funds:

Current: Accounts Payable:			
Committee C-1	$202.20		
Advance Collections	5 229.96		
Total Current Liabilities—General Fund		$5 432.16	
Life Membership Fund	3 295.20		
Headquarters Fund	6 491.56		
Publication Fund	2 515.00		
Book of Standards Reserve	15 000.00		
Executive Retirement Reserve	7 780.00		
Retirement Fund Reserve	3 425.00		
Reserve for Depreciation of Investments	12 000.00		
Joint A.I.M.E.-A.S.T.M. Publication Fund	475.73		
Surplus	165 037.47		
		216 019.96	
Total Liabilities General Funds			$221 452.12

Building Fund:

Contributions	$144 998.43		
Accumulated Income on Invested Funds	257.72		
Mortgage Payable, dated December 23, 1943, due within 5 years with quarterly installments of $187.50 at 5 per cent $23 875.00 reduced to	23 500.00		
Total Building Fund			168 756.15

Special and Designated Funds:

A.S.T.M. Research Fund:			
Principal	$40 281.49		
Income	3 548.11	$43 829.60	
Dudley Medal and Marburg Lecture Fund:			
Principal	6 625.00		
Income	409.43	7 034.43	
Richard L. Templin Award Fund:			
Principal	1 000.00		
Income	112.50	1 112.50	
Committee Funds, unexpended balances		24 461.23	
Total Special and Designated Funds			$76 437.76
Total Liabilities and Funds			**$466 646.03**

COMPARISON OF GENERAL FUNDS FOR FISCAL YEARS 1941–1945 INCLUSIVE.

At Close of Fiscal Year	Assets				Liabilities			
	Cash	Investments	Accounts Receivable	Furniture and Fixtures	Accounts Payable	Reserve for Book of Standards	Miscellaneous Funds	Surplus
1941	$18 206.61	$88 001.45	$14 120.99	$6440.58	$3937.44	$13 890.25	$12 907.43	$96 034.51
1942	14 915.67	93 281.45	6 846.09	6362.86	5036.28	13 890.25	17 902.43	84 577.11
1943	29 220.41	111 136.68	14 567.65	6960.38	5569.02	17 113.98	33 541.26	105 660.86
1944	42 637.45	114 237.61	11 986.97	6780.66	9009.15	17 113.98	37 913.05	111 606.51
1945	46 058.60	153 718.05	15 033.90	6641.57	5432.16	15 000.00	35 982.49	165 037.47

RECEIPTS AND DISBURSEMENTS

FOR THE PERIOD ENDED DECEMBER 31, 1945

Cash balance, January 1, 1945			$42 637.45

RECEIPTS

Operating Receipts (Budgeted):			
Dues and Entrance Fees:			
Current Dues	$118 249.65		
Past Dues	1 006.40		
Advance Dues	2 683.70		
Income, Life Membership Fund	150.00		
Entrance Fees	4 645.00		
Total Dues		$126 734.75	
Sale of Publications:			
Book of Standards (Members addl. parts)	$15 777.70		
1942 Book of Standards and Supplements	1 377.76		
1944 Book of Standards	72 714.31		
1945 Supplements, Book of Standards	209.70		
Methods of Chemical Analysis of Metals	1 851.22		
Compilations of Standards	28 886.01		
Separate Standards	8 113.06		
Selected Standards for Students	804.20		
Proceedings	4 534.33		
Spring Meeting Papers	2 004.09		
Bulletin Subscriptions (non-members)	648.45		
Special & Miscellaneous Publications	39 448.65		
Total Sale of Publications		$176 369.48	
Miscellaneous:			
Advertising, Bulletin	$12 181.63		
Advertising, Index	1 896.50		
Interest and Dividends	6 019.08		
Committee A-1 for Technical Assistant	708.87		
Miscellaneous	154.87		
Total Miscellaneous Items		$20 960.95	
Total Operating Receipts (Budgeted)		$324 065.18	
Nonoperating Receipts (Not Budgeted):			
Investments Matured or Sold	$19 563.00		
Excess Remittances	538.71		
Committee C-1 for Technical Assistant	1 700.00		
Executive Retirement Reserve	225.00		
Total Nonoperating Receipts		$22 026.71	
Total Receipts			$346 091.89
Total Receipts and Cash Balance, January 1, 1945			$388 729.34

Brought Forward			**$388 729.34**

DISBURSEMENTS

Operating Disbursements (Budgeted):			
Publications:			
Year Book	$4 777.38		
1944 Book of Standards	5 696.06		
1945 Supplements, Book of Standards	20 282.61[a]		
Emergency Alternate Provisions	761.47		
Compilations of Standards	8 930.88		
Separate Standards	5 313.98		
Chemical Analysis of Metals	89.31		
Proceedings	16 526.05[a]		
Preprints	3 545.38		
Spring Meeting Papers	18.83		
A.S.T.M. Bulletin	12 009.71		
Circulars to Members	2 196.66		
Index to Standards	4 900.39		
Special and Miscellaneous Publications	14 410.94		
Authors Reprints, Electros, etc. at Cost	1 202.31		
Total Disbursements, Publications		$100 661.96	
Salaries		99 090.40	
General Office Expenses		22 185.09	
Expenses, Technical and District Committees		3 957.54	
Expenses, Meetings		6 148.78	
American Standards Assn.		1 000.00	
Traveling Expenses, Administrative & Special Committees		5 369.00	
Rent, Headquarters		8 934.48	
Operating Expenses—New Headquarters		1 564.20	
Furniture and Fixtures		809.71	
Principal A.S.T.M. Research Fund		2 300.00	
Dues, Contributions, Miscellaneous		374.50	
Legal Services		1 750.00	
Employees Retirement Fund		8 045.32	
Total Operating Disbursements (Budgeted)		$262 190.98	
Nonoperating Disbursements (Not Budgeted):			
Refund of Excess Remittances	$538.71		
Investments	59 512.94		
Accrued Interest	200.89		
Book of Standards Reserve	17 113.98		
Technical Assistant Committee C-1	1 667.68		
Joint A.I.M.E.-A.S.T.M. Publication Fund	1 445.56		
Total Nonoperating Disbursements		$80 479.76	
Total Disbursements			$342 670.74
Cash Balance, December 31, 1945			$46 058.60

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[a] These amounts include a check for $33,900. drawn against the cost of the 1945 Supplements to the Book of Standards, and the 1945 *Proceedings*, but not actually paid on December 31, 1945.

A.S.T.M. BUILDING FUND

DETAILS OF BUILDING FUND CASH RECEIPTS AND DISBURSEMENTS FOR THE PERIOD JANUARY 1, 1945 TO DECEMBER 31, 1945

Receipts

Total Contributions		$144 998.43	
Income from Investments:			
Interest on U. S. Treasury Bonds 2½s 1967–72		312.50	$145 310.93

Disbursements

Purchase of $25 000 U. S. Treasury Bonds 2½s 1967–72 at $101.16 dated 6/1/45, July 3, 1945	$25 390.63		
plus accrued interest	54.78	$25 445.41	
Purchase of premises 1916–18 Race St., Philadelphia, Pa. per settlement certificate No. 934259 of the Commonwealth Title Co. of Philadelphia dated August 1, 1945.			
Purchase Price	$53 625.00		
Commission to Agent	2 681.25		
Settlement fees, net	197.10		
	56 503.35		
Less, mortgage assumed	$23 875.00	$32 628.35	
Rent paid June, 1945, to facilitate possession	110.00		
Zoning fees and permits	17.50	127.50	
Architect's fees		1 200.00	
Reduction of Mortgage—2 quarterly payments of $187.50 each		375.00	59 776.26
Cash Balance, December 31, 1945			$85 534.67

DETAIL OF ASSETS AND LIABILITIES OF BUILDING FUND, AS OF DECEMBER, 31, 1945

Assets

Cash in Girard Trust Co., Philadelphia, Pa.		$85 534.67	
Investments $25 000. U. S. Treasury Bonds 2½s 1967–72 at $101.16. (Market Value $25 367.19)—Cost		25 390.63	
Accounts Receivable:			
Sales and salvage of equipment at 1916–18 Race St., Philadelphia, Pa. incident to possession of property and preparation for alterations—Net of expenses		466.69	
Property, 1916–18 Race St., Philadelphia, Pa.			
Purchase price	$53 625.00		
Commission to Agent	2 681.25		
Settlement fees, net	197.10		
	56 503.35		
Allocated to:			
Land		30 000.00	
Building		26 503.35	
Construction Costs:			
Architect's fee	$1 200.00		
Rent paid to facilitate possession	110.00		
Zoning fees and permits	17.50		
	1 327.50		
Less, salvage sales, as above	466.69	860.81	
Total Assets			$168 756.15

Liabilities

Contributions		144 998.43	
Income on Investments:			
6 months interest to 12/1/45 on U. S. Treasury Bonds 2½s 1967–72	312.50		
Less, accrued interest at purchase	54.78	257.72	
Mortgage Payable—dated Dec. 23, 1943 due within 5 years with quarterly installments of $187.50 at 5 per cent	23 875.00		
Less, 2 quarterly payments of $187.50 each	375.00	23 500.00	
			$168 756.15

In addition to the General Funds and Building Fund, the Society also had on hand the following funds, as of December 31, 1945:

A.S.T.M. RESEARCH FUND

Principal Account:	*Total*	*Invested*	*Uninvested Cash*
Balance January 1, 1945	$39 198.49	$37 408.99	$1 789.50
Receipts:			
Contribution—A.S.T.M. Current Entrance Fees	+2 300.00		+2 300.00
Sale of Securities		−2 293.00	+2 293.00
Loss on Securities sold or called	−1 217.00	−1 217.00	
	$40 281.49	$33 898.99	$6 382.50
Disbursements:			
Purchase of Securities		+4 787.25	−4 787.25
Balance, December 31, 1945	$40 281.49	$38 686.24	$1 595.25
Income Account:			
Balance January 1, 1945	$2 674.19		
Receipts:			
Interest and Dividends on Investments	1 173.92		$3 848.11
Disbursements:			
Engineering Foundation			300.00
Cash Balance December 31, 1945			$3 548.11

DUDLEY MEDAL AND MARBURG LECTURE FUND

Balance January 1, 1945			
Principal—Investments (at cost)	$6 625.00		
Income—Cash	343.43	$6 968.43	
Receipts			
Interest on Investments		66.00	$7 034.43
Disbursements			
None			
			$7 034.43
Balance, December 31, 1945:			
Principal—Investments (at cost)		$6 625.00	
Income—Cash		409.43	$7 034.43

RICHARD L. TEMPLIN AWARD FUND

Principal—Investments (at cost)	$1 000.00	
Receipts		
Contributions from A.S.T.M. for Cost of Certificate	100.00	
Interest on Investments	12.50	$1 112.50
Disbursements		
None		
		$1 112.50
Balance, December 31, 1945:		
Principal—Investment (at cost)	1 000.00	
Income—Cash	112.50	$1 112.50

COMMITTEE FUNDS

Committee A-1 on Steel	$1 213.72	
Committee A-5 on Corrosion of Iron and Steel	3 202.42	
Committee B-3 on Corrosion of Non-ferrous Metals and Alloys	2 598.44	
Committee B-6 on Die Cast Metals and Alloys	2 456.68	
Committee B-8 on Electrodeposited Metallic Coatings	45.36	
Committee C-1 on Cement	840.76	
Committee C-1 on Master Builders Co. Special Fund	6 916.51	
Committee C-1 —Cement Reference Laboratory	500.00	
Committee C-9 on Concrete and Concrete Aggregates	200.82	
Committee C-9 on Sanford E. Thompson Medal Fund	403.25	
Committee C-15 on Manufactured Masonry Units	366.56	
Committee C-18 on Natural Building Stones and Slate	176.36	
Committee D-1 on Paint, Varnish, Lacquer, and Related Products	74.25	
Committee D-2 on Petroleum Products and Lubricants	167.01	
Committee D-5 on Coal and Coke	1 357.77	
Committee D-12 on Soaps and Other Detergents	73.30	
Committee D-13 on Textile Materials	339.46	
Committee D-13 on Research Textile Finishes	238.34	
A.S.A. Sectional Committee on Specifications for Cast Iron Pipe and Special Castings	1 741.77	
Research Committee on Fatigue of Metals	197.56	
Detroit District Committee	1 132.00	
Philadelphia District Committee	179.57	
Pittsburgh District Committee	55.50	
Total Committee Funds		$24 477.41
Accounted for as follows:		
Cash Balance, December 31, 1945	$15 461.23	
Investments—at cost	9 000.00	
Accrued interest on bonds purchased	16.18	
		$24 477.41

INVESTMENTS

AS OF DECEMBER 31, 1945

GENERAL FUNDS

Number of Shares or Par Value		*Cost or Book Value*	*Marke Value*
	Bonds		
$2 000	Associated Electric Co. 4½'s, due 1953	$1 693.50	$2 035.00
2 000	Columbia Gas & Electric Co. Debentures 5's, due 1961	2 025.34	2 090.00
4 000	Erie R. R. Co. General Mortgage, Income 4½'s, due 2015	2 832.00	3 920.00
2 000	Hudson Coal Co. S. F. 5's Series A, due 1962	1 697.25	1 600.00
3 000	Southern Pacific Debentures 4½'s, due 1969	2 544.75	3 090.00
13 000	U. S. Savings Bonds, Series G 2½'s[c]	13 000.00	12 370.00
4 800	U. S. Treasury Bonds, 2½'s, due 1967–72	4 800.00	4 870.50
2 000	U. S. Treasury Bonds, 2½'s, due 1964–69	2 000.00	2 051.45
2 000	U. S. Treasury Bonds, 2's, due 1952–54	2 000.00	2 088.12
50 000	U. S. Treasury Bonds, 2's, due 1952–54	50 765.63	52 203.12
22 000	U. S. Treasury Notes, 1¼'s, due March 1947	22 000.00	22 082.50
		$105 358.47	$108 400.49
	Stock		
19	American Cyanamid Co., 5 per cent cumulative preference	190.00	218.50
25	Colgate-Palmolive-Peet Co., $3.50 preferred	2 525.00	2 650.00
50	General Telephone Corp., $2.50 preferred[a]	2 675.00	3 450.00
20	G. C. Murphy Co., 4¾ per cent preferred	2 100.00	2 290.00
100	Murray Corp. of America, 4 per cent cumulative preference	5 050.06	5 025.00
75	Allis-Chalmers Mfg. Co., common, no par value	3 316.20	3 993.75
25	American Can Co., common	2 122.30	2 512.50
50	American Cyanamid Co., Class B common	1 764.25	2 425.00
100	American Rad. & Std. Sanitary Corp., common	1 564.32	1 825.00
25	American Tobacco Co., common	2 110.12	2 225.00
100	Atlantic Refining Co., common par $25	2 302.50	3 950.00
10	Delaware Power & Light Co., common	135.00	240.00
25	Hazel Atlas Glass Co.	2 510.64	3 056.25
100	S. S. Kresge Co., common	2 490.18	3 300.00
100	Mathieson Alkali Works, common, no par value	3 016.00	3 150.00
25	J. C. Penney Co., common	2 279.63	3 721.88
67	Philadelphia Electric Co., common	1 185.06	1 909.50
116	Public Service Corp. of N. J., common	4 369.50	2 813.00
50	The Texas Company	2 519.55	3 050.00
20	United Gas Improvement Co., common	315.00	477.50
50	U. S. Gypsum, common	3 819.27	5 700.00
		$48 359.58	$57 982.88
	TOTALS	$153 718.05	$166 383.37

A.S.T.M. BUILDING FUND

Number of Shares or Par Value		*Cost or Book Value*	*Market Value*
	Bonds		
$25 000	U. S. Treasury Bonds, 2½'s, 1967–72........	$25 390.63	$25 367.19

A.S.T.M. RESEARCH FUND

Number of Shares or Par Value		*Cost or Book Value*	*Market Value*
	Bonds		
$2 000	Associated Electric Co., 4½'s, due 1953......	$1 693.50	$2 035.00
1 500	Balto. & Ohio R. R. General and Refunding 5's series F, due 1996..................	1 511.25	1 436.25
2 000	Columbia Gas & Electric Co., Debentures 5's, due 1961............................	2 025.31	2 090.00
2 000	Hudson Coal Co. S. F., 5's, Series A, due 1962	1 697.25	1 600.00
4 500	U. S. Defense Savings Bonds, series G, 2½'s[c]..	4 500.00	4 278.00
2 000	U. S. Treasury Bonds, 1965–70, 2½'s.........	2 000.00	2 046.87
3 000	U. S. Treasury Notes, 1¼'s, March, 1947....	3 000.00	3 011.25
		$16 427.31	$16 497.37
	Stock		
30	Butler Bros., cumulative preferred..........	3 090.00	3 270.00
12	Consolidated Edison Co. of N. Y. $5 preferred..................................	1 234.39	1 296.00
29	Consumers Power Co., $4.50 preferred, no par value............................	2 914.50	3 284.25
30	G. C. Murphy Co., 4¾ per cent preferred.....	3 150.00	3 435.00
100	Allis-Chalmers Mfg. Co. common, no par value..................................	4 421.59	5 325.00
5	Delaware Power & Light Co., common.....	67.50	120.00
98	General Motors Corp., common, par $10[b]....	6 504.75	7 399.00
33	Philadelphia Electric Co., common.........	583.70	940.50
9	Public Service Corp. of N. J., common......	135.00	218.25
10	United Gas Improvement Co., common.....	157.50	238.75
		$22 258.93	$25 526.75
		$38 686.24	$42 024.12

DUDLEY MEDAL AND MARBURG LECTURE FUND

Number of Shares or Par Value		*Cost or Book Value*	*Market Value*
$ 500	Balto. & Ohio R. R. Refunding & General Mortgage, 6's, Series C, due 1995.........	$ 550.00	$ 508.13
6 000	Balto. & Ohio R. R. Refunding & General Mortgage, 5's, Series A, 1995..............	6 075.00	5 812.50
		$6 625.00	$6 320.63

RICHARD L. TEMPLIN AWARD FUND

Number of Shares or Par Value		*Cost or Book Value*	*Market Value*
$1 000	U. S. Treasury Bonds, 2½'s, 1967–72.........	$1 000.00	$1 014.68

COMMITTEE FUNDS

Number of Shares or Par Value		*Cost or Book Value*	*Market Value*
1 000	U. S. Treasury Bonds, 2⅞'s, 1960–55.........	$1 000.00	$1 148.44
2 000	U. S. Treasury Bonds, 2½'s, 1964–69.........	2 000.00	2 051.25
3 000	U. S. Treasury Bonds, 2½'s, 1965–70.........	3 000.00	3 070.31
3 000	U. S. Treasury Bonds, 2½'s, 1967–72.........	3 000.00	3 044.06
		$9 000.00	$9 314.06
	GRAND TOTAL.........................	$234 419.92	$250 424.05

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[a] Market values were taken from current financial publications with the exception of quotations on inactive or unlisted securities indicated by footnote *a*. These values were obtained from brokers as of December 31, 1945.

[b] The income from these shares is, by agreement, paid to an annuitant.

[c] Market value is stated at redemption value as of December 31, 1945.

and supplies; and second, adjustment of the Society's wage and salary scales to present economic trends and conditions. Making adequate provision for these factors and providing for such staff expansion as seems appropriate at this time, it has been estimated that disbursements for 1946 will be $348,500, which provides for meeting the full costs of printing the 1946 Book of Standards from current funds and the established reserve of $15,000.

In estimating current receipts, the Executive Committee felt that while there was no indication at the beginning of the year of any serious reversal of trend in growth of Society income, there is obviously much economic uncertainty and adverse conditions could develop at any time. Accordingly, it has been considered wise to be definitely on the conservative side and assume that there may well be a falling off in the *rate* of income from membership dues, sales of publications and miscellaneous sources. Current receipts have therefore been conservatively estimated at $297,000. To balance the budget the sum of $51,500 will if necessary be applied from the reserve of the Society as follows: $2000 from the Publication Reserve to be applied to costs of committee work in preparation of X-ray Diffraction Data Cards; the Headquarters Reserve of approximately $6500 to be applied to the cost of moving Society headquarters and purchase of new furnishings; and $43,000 from net surplus.

This application of reserve funds to finance expansion of Society activities and to provide funds for operations in the "off year" of receipts from Book of Standards sales is in line with established policy. To a certain extent this use of such funds is equivalent to a "pump priming" operation to meet initial costs of new activities, which in time will pay their way through increased membership and new publications.

A new feature of the 1946 budget is a section on Headquarters Occupancy Expense which provides for estimated expenses of headquarters occupancy substantially equivalent to the item of rent in previous budgets. Included in this section of the budget are such items as taxes, interest on mortgage, cost of utility services (electricity, water, steam), other operational costs such as janitorial, equipment and cleaning services, and maintenance and repairs. This section will also include the charge for depreciation of building and equipment previously mentioned.

The budget was prepared in January. A careful review in April revealed no need for any changes. The Executive Committee will further review all financial operations during the annual meeting.

In January, 1946, the Executive Committee transferred from the net surplus reported on December 31, 1945, the following sums to the accounts indicated: $7000 to the Publication Reserve, being a substantial part of the net profits on sale of X-ray Diffraction Data Cards, to be used to support the work of the Joint Committee on Chemical Analysis by X-ray Diffraction Methods in the development of further data and publication thereof; $3500 to the AIME-A.S.T.M. Publication Fund to meet the cost of the Joint Symposium on Stress Corrosion Cracking, matching a similar contribution of the AIME; and $2000 to the Executive Retirement Reserve.

Since January 1, 1946, there has been sold from the General Funds $22,000 of U. S. Treasury Notes $1\frac{1}{4}$ due March, 1947, yielding $22,097.45. Investments have been made in those funds in the sum of $26,678.11, as follows: 100 shares of Baldwin Locomotive, 100 shares of International Harvester, 100 shares of Carpenter Steel, 30 shares of American Viscose, and $5000 Convertible Bonds $3\frac{1}{4}$ of the Walworth Co. Cash in the principal of the Research Fund was invested in 100 shares of Ohio Oil, in the amount of $2167.88.

The total book value of Society investments as of May 31, 1946, in all funds was $241,265.91. The Society's cash balance on May 31, 1946, was $159,995.87.

Retirement Plan for Employees:

The Retirement Plan for Employees has been continued upon the basis established in May, 1944, which was described in the Executive Committee report for that year.[7] During the past year, three employees in the Plan have resigned from the staff and one employee has joined the Plan, making 19 of 26 eligible employees now in the Plan.

The Society's total contribution in premiums for retirement policies for the above-mentioned 19 employees is at the annual rate of approximately $8350, which is roughly 7 per cent of the present salary roll and 2½ per cent of estimated current disbursements. The total of employees' annual contributions, including insurance benefits which most of them have elected, is approximately $2450.

Cooperative Activities:

Discussions have been continued during the year with various government agencies looking towards better collaboration between these agencies and the Society in the development of government and A.S.T.M. specifications. The organization for development of Federal specifications has been substantially revised by the Government through the formation of the Federal Specifications Board (to replace the former Federal Specifications Executive Committee) and through strengthening the Specifications Branch of the Procurement Division, Treasury Department. The Executive Committee had the opportunity to review the plans for reorganization and submitted several comments and recommendations to the Director of Procurement. The new set-up should greatly facilitate and expedite development and revision of Federal specifications and at the same time provide the means of more direct cooperation between the technical specification committees of the Federal Specifications Board and committees of A.S.T.M. Provision is being made for direct contact of staff members of F.S.B. and A.S.T.M., which it is believed will help to keep Federal and A.S.T.M. specification activities in step one with the other.

An important feature of the new organization for Federal specifications is the establishment of an Industry Advisory Council, the members of which are appointed by the Secretary of the Treasury from industry and from various technical organizations dealing with the development of specifications and standards. The purpose of this Council is to provide closer relationship between industry and technical societies and the Federal Specifications Board, to bring about the maximum utilization of and conformity with industrial practice, to promote close cooperation of industry suppliers and manufacturers, and to obtain the fullest technical participation and assistance of industry in the development of Federal specifications. Provision is being made for the establishment of industry task committees wherever these would be advantageous, to assist in the development of Federal specifications. Use will also be made of committees of technical societies as advisory task groups where the representation is adequate for this purpose. The Executive Secretary has been named as a member of the Industry Advisory Council.

Application of these principles of cooperation was made early this year through the establishment of an A.S.

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[7] *Proceedings*, Am. Soc. Testing Mats., Vol. 44, p. 59 (1944).

T.M. conference group to advise with the Federal Technical Committee on Liquid Fuels concerning proposed Government Specifications for Motor Fuel. A joint meeting of the A.S.T.M. conferees with representatives of the Federal committee was held in Washington on February 15, at which proposed Government specifications were discussed in detail and recommendations of representative members of the petroleum and automotive industries were submitted to the committee. The conference group has been retained in anticipation of periodic meetings for further review of the specifications.

The Executive Committee has continued to urge upon all Federal specification agencies the importance of their direct and active participation in A.S.T.M. committee work on specifications and methods of test. Generally speaking the gains in such representation and participation made during the war have been retained, although with demobilization of the Armed Services there have naturally been many changes in representation. Such direct participation in Society committee work is of inestimable value both to the government and the Society in their respective specification activities, and through it there continue to be numerous examples of close coordination between the work of A.S.T.M. and Federal specifications committees.

Another phase of Government specification activities in which the Society and many of its committees are greatly interested is that represented by the work of the Joint Army-Navy Specification Board, which deals primarily with the harmonizing of requirements of specifications of the Army and Navy for military materiel. The work of this Board has resulted in many joint Army-Navy (JAN) specifications, which form an increasingly important group of government specifications where coordination with industry specifications, including those of A.S.T.M., is important. Informal discussions have been continued during the year on the establishment of procedures whereby the Society can render an effective service to the JAN Board, especially in advising on tests and specification requirements for materials and products that are produced for both military and industrial applications. Several lines of cooperation have been established, especially in the fields of electrical insulating materials and plastics.

The Society's cooperative relations with its sister technical organizations have been continued during the year along lines of well-established policy. The formation of certain new joint committees and several Society appointments to committees of other organizations are noted below.

The Executive Committee accepted an invitation from the American Welding Society to appoint representatives to a Joint Conference Committee of the A.W.S., A.S.T.M., and the American Iron and Steel Institute to consider specifications for structural steel for welding. Messrs. David Arnott, American Bureau of Shipping, N. L. Mochel, Westinghouse Electric Corp. and chairman of Committee A-1 on Steel, and A. R. Wilson, Pennsylvania Railroad Co., were named as the Society's representatives on the conference committee.

V. J. Altieri, Eastern Gas and Fuel Associates, has been named as the Society's representative on the Chemical Section of the American Gas Association.

R. W. Lahey, American Cyanamid Co., was appointed as the Society's representative on the Container Testing Committee of the Technical Association of the Pulp and Paper Industry.

E. R. Young, Climax Molybdenum Corp., was appointed as representative of Committee A-3 on Cast Iron on the

Specification Study Committee of the Gray Iron Foundry Division, American Foundrymen's Association.

The Executive Secretary has accepted membership on a Joint Committee on Measurement of Opinion, Attitudes and Consumer Wants, sponsored by the National Research Council and the Social Science Research Council. The work of this joint committee is expected to be of value to the Society's work in the field of ultimate consumer goods in that establishment of adequate methods of determining consumer wants should assume increasing importance as work in that field develops.

American Standards Association:

The most important development this year in the affairs of the American Standards Association, and of much significance to its member bodies, is the attention that has been given to the rôles of Government and industry in standardization activities. The conference of industrial executives held in January, 1945, at the call of the Secretary of Commerce (mentioned in the annual report last year) led to the formation of a Policy Committee on Standards under the chairmanship of Charles E. Wilson, President of the General Electric Co. This committee in June, 1945, presented a report to the Secretary of Commerce which (1) advocated that industry should assume leadership in the work of industrial standardization and that "standards activities which involve negotiation, opinion, judgment or compromise should be developed through individual and joint efforts of technical, manufacturing, merchandising and consumer groups"; (2) expressed the belief that the rôle of Government should be essentially that of research to provide data for standards and economic and marketing information; and (3) recommended that industrial standardization should be coordinated and promoted through the American Standards Association as the federation of the standardizing bodies of the country. The report also discussed the work of the Department of Commerce in the development of trade standards and made certain recommendations relating thereto, which in effect call for a functional division of effort between the Department of Commerce (including the National Bureau of Standards) and interested private groups (including the American Standards Association) and for cooperation between all parties concerned. The Policy Committee Report is published in full in *Industrial Standardization*, July, 1945.

The Executive Committee found itself very generally in agreement with the report in principle and lent its support thereto in the counsels of ASA. While not agreeing necessarily with all statements in the supporting memorandum to the report, the Executive Committee particularly endorsed the statement therein defining a field for ASA and the following statement that appears as a conclusion to the memorandum:

> "A digest of this supporting memorandum clearly demonstrates to this committee that it is to the forward-looking self-interest of business that its executives should make adequate provision for the handling of standardization problems, first, within their own companies, and second, within their trade associations, and thirdly, within the professional and technical societies. It is equally to their interest that they should, through their companies and trade associations, adequately support, both with effort and funds, not only the standardization work of the national, professional, technical and trade associations in which they have or should have a part, but also, through affiliation, activity and the supply of adequate finances, support the work of the federation of these bodies—the American Standards Association."

Under date of February 28, 1946, Secretary of Commerce Wallace addressed a letter to Mr. Wilson which set forth the views of the Department of Commerce. The letter is published in *Industrial Standardization*, April, 1946.

In brief, Mr. Wallace expressed agreement with the main thesis of the Policy Committee Report, saying that the Department could accept the general objectives and spirit of the report and would be glad to see the American Standards Association and other organizations pursue a vigorous program in the field of trade standards. He gave assurance of willingness of the Department to cooperate to the fullest extent in providing scientific, technical, economic and marketing data useful in such a program. However, he reserved to the Department of Commerce its present work of developing trade standards at the request of industry, explaining that the Department has a statutory responsibility to provide such services in the interest of business and industry and the general public. At the same time he indicated the intention to modify the rôle in this work of the National Bureau of Standards. Finally, he said that to the extent that ASA is able to perform trade standard services to the satisfaction of all groups concerned, the Department is prepared to encourage the use of ASA facilities for the initiation, development and publication of standards.

The Policy Committee on Standards has been asked to continue to function as an advisory group to the Department of Commerce, and a joint conference committee of the ASA and the Department has been established to facilitate cooperative activities.

These matters are of obvious importance to this Society, which for nearly fifty years has been engaged in the development of standards for tests and specifications for materials. We have an obvious stake in the successful development of such work in the future, and in the establishment of the most effective relation between industry and Government in relation thereto. Accordingly, the Executive Committee is giving the fullest consideration to these developments, and at the moment is engaged in discussion of salient points with the officers of ASA.

The amendment of the Constitution of the American Standards Association referred to in the report a year ago and for which the Executive Committee voted affirmatively for the Society has been adopted, including the statement of basic principles proposed by A.S.T.M. The main effect of the amendment of the Constitution is to broaden the scope of ASA to cover standardization in other fields than those of strictly engineering.

In its report a year ago the Executive Committee referred to a proposal that had been announced for incorporation of the American Standards Association through a Federal charter and to the fact that the Executive Committee was strongly opposed to such incorporation under the particular terms proposed. A conference of representatives of member bodies of ASA was held in September, 1945, at which the subject was thoroughly discussed. As a result of that conference the Board of Directors of ASA enlarged its Committee on Congressional Charter by the appointment of representatives of the Society of Automotive Engineers, National Lumber Manufacturers Association, and this Society. In accordance with this action the Executive Secretary accepted membership on the enlarged Charter Committee and has participated in three meetings of the committee. A proposal for State incorporation has been advanced and the questions at issue are still under consideration.

The Conference of Staff Executives of Member Bodies of ASA, which as noted a year ago was established by appointment of the ASA Board of Directors, has begun to function. The first meeting of the Conference was held on March 7, 1946, at which the legal aspects of standardization were ably discussed by Mr. James V. Hayes; and Mr. Howard Coonley, newly appointed chairman of the ASA Executive Committee, addressed the members of the Conference on recent developments in ASA with particular reference to the relationships with the Department of Commerce previously discussed. The Executive Secretary of the Society is serving as chairman of the Conference.

J. R. Townsend has been re-appointed as one of the Society's representatives on the ASA Standards Council for a term of three years. R. P. Miller has been re-appointed as the Society's representative on the ASA Building Code Correlating Committee for a term of two years.

International Activities:

The Society has continued work on proposed international standardization projects cleared through the United Nations Standards Coördinating Committee, in which the American Standards Association is the representative of the United States. Responsibility for direction of this work has been delegated by the Executive Committee to the Administrative Committee on Standards and reference to several UNSCC projects will be found in the appended report of that committee.

In October, 1945, there was held in New York City an International Conference under the auspices of the United Nations Standards Coordinating Committee. The purpose of the conference was to consider steps that should be taken towards the formation of a new international standards association and to undertake the development of a proposed constitution for such an organization. To facilitate American participation in this conference, the president of the American Standards Association appointed a temporary ASA Committee on International Standardization Work, in which the A.S.T.M. as a member body of ASA was represented by the Executive Secretary. This committee examined a preliminary draft of a constitution and three of its members were named as advisers to the American delegates to the conference. The conference was successful in its deliberations and adopted a constitution for submission to those national standards bodies who express a desire to participate in the formation of the new international standards organization. At the moment means of formally disbanding the old International Standards Association are under consideration and plans are being laid for a further conference in London next fall, at which formal organization of the new body may be consummated.

The organization of an international association is related in an important manner to the establishment under the United Nations Charter of the Economic and Social Council, whose purposes are so broadly defined as to permit of including international standardization within its scope. The Council is authorized to consult non-governmental organizations that are concerned with matters within its competence, so that the existence of an International Standards Association organized by the national standards bodies of the world is highly desirable.

Accordingly, the Executive Committee favors the formation of a new International Standards Association upon which the ASA will be the American member, provided that procedures will be established whereby international standardization projects will be channeled into the appropriate ASA member bodies for study and development from the American standpoint.

It is understood that the United Nations Standards Coordinating Committee will carry on its activities until the formation of the international standards association, after which it will be disbanded and its work turned over to the new organization.

There have been a number of interesting contacts during the year with standardizing and technical bodies of other nations. Representatives of such bodies from Great Britain, Australia, New Zealand, Norway, Denmark, Holland, France, Belgium, China, Brazil, Uraguay and Chile visited the Society headquarters and conferred at length with various members of the staff. Resumption of normal relations and interchange of standards is greatly to be desired, and the staff is giving particular attention to all inquiries from foreign organizations. These international relationships are also being strengthened by the return to membership of many of our foreign members and the acquisition of new members from abroad.

Respectfully submitted on behalf of the Executive Committee,

J. R. TOWNSEND,
President.

C. L. WARWICK,
Executive Secretary.
June 24, 1946.

APPENDIX I

REPORT OF ADMINISTRATIVE COMMITTEE ON STANDARDS

Since the 1945 Annual Report of the Committee on Standards (then designated Committee E-10) an unusually large number of recommendations has been submitted to the committee and received consideration. As indicated in that report, rather than to have the recommendations dealing with new tentatives and revisions of tentatives submitted at the annual meeting, these were submitted instead to the Committee on Standards. A special meeting for consideration of these recommendations was held on June 27, 1945. As a result of action taken at this meeting, 39 new tentatives were accepted for publication as well as 42 revisions of tentatives and 3 tentative revisions of standards. These are recorded in the Summary of Proceedings, 1945.[1]

Several of the recommendations appearing in the annual reports were carried over, since the information included on the letter ballot vote was incomplete. These, together with a number of new recommendations, then came up for consideration at a meeting of the Committee on Standards held on August 27. As a result of the action taken, 14 new tentatives were accepted as well as 7 revisions of tentatives, 10 tentative revisions of standards, the revision of an Emergency Alternate, the withdrawal of an Emergency Standard, and the withdrawal of a tentative and a standard. A list of these appears in Appendix A attached to this report. Since then more recommendations have been acted upon by letter. These are listed in Appendix B.

Actions Under Emergency Procedure:

The following two recommendations other than those acted upon at the meeting of August 27 referred to above, have been received and acted upon during the year:

ES-43a	Emergency Method of Test for Sulfated Residue from Lubricating Oils by Air Ignition.
EA-D 119a	Specification for Rubber Insulating Tape. This revision was later withdrawn.

As might well be expected, many of the Emergency Alternates and Emergency Specifications are now being discontinued on the recommendation of the technical committees concerned.

Emergency Alternates have been withdrawn on the recommendation of the technical committee concerned as follows:

	Submitted by:	
EA-D 97	D-2	Method of Test for Cloud and Pour Points.
EA-D 524	D-2	Method of Test for Carbon Residue of Petroleum Products (Ramsbottom Carbon Residue).
EA-D 147	D-8	Methods of Testing Bituminous Mastics, Grouts and Like Mixtures.
EA-D 353	D-11	Specifications for Insulated Wire and Cable: Performance Rubber Compound.
EA-D 469a	D-11	Specifications for Insulated

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[1] Summary of Proceedings of the Forty-Eighth Annual Meeting, *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 1 (1945).

		Wire and Cable: Heat-Resisting Rubber Compound.
EA-D 532a	D-11	Tentative Specifications for Rubber Sheath Compound for Electrical Insulated Cords and Cables.

The Emergency Specifications ES-31 have been withdrawn and replaced by Tentative Specifications for Electrodeposited Coatings of Lead on Steel (B 200 – 45 T).

All of the Emergency Alternates and Specifications that are still in force are receiving attention in the technical committees, particularly since it has been proposed by the Executive Committee that all such Emergency Standards and Alternates be discontinued by the close of the Annual Meeting. While in most instances, there will be no further need of the emergency recommendations, nevertheless conditions continue to exist in certain fields which will necessitate special provisions being made in the standards in question, as for example those in the soap field.

Expansion of Standardization Activities:

At no time in the Society's history has there been such expansion taking place in the technical committee activities. New committees are in process of organization or in prospect on the following subjects:

Asbestos-Cement Products.—A personnel has been developed and acceptances received so that a meeting to effect organization will be held in the near future.

Oxychloride Cement.—The personnel of a proposed committee authorized at the October meeting of the Executive Committee is now being assembled. The function of this committee is to develop test procedures and specifications for oxychloride cements for use in connection with flooring, molds, refractories, etc. It should be of direct assistance to a new sectional committee on oxychloride cement flooring which is being organized under the joint sponsorship of the A.S.T.M. and the National Bureau of Standards.

Engine Antifreezes.—A special study committee which was organized to review the field is recommending to the Executive Committee an outline of scope and proposed personnel for a new technical committee on engine antifreezes. Upon the approval of the Executive Committee, the organization of this committee may be completed prior to the annual meeting.

Sulfur Cements.—The Society still has before it the organization of a committee on sulfur cements authorized several years ago, but the organization of which was held up in view of wartime conditions.

Cutting Oils.—The Committee on Standards referred to Committee D-2 on Petroleum Products and Lubricants the question of organizing work on cutting oils, and Committee D-2 is now setting up a new technical committee to handle the subject.

Other Subjects Requiring Development.—Two fields that the Committee on Standards would be interested in seeing developed are those of leather for cushioning, belting, gaskets, etc., and of porcelain enamels. Particularly in the latter field, in view of inquiries that are received, it would appear that the subject would justify development in the A.S.T.M.

Submission of Standards to the A.S.A.:

The submission of A.S.T.M. standards to the A.S.A. for approval as American Standard has been confined for the most part to standards under the jurisdiction of sectional committees:

Sectional Committee Z11:

Test for Carbonizable Substance in Paraffin Wax (A.S.T.M. D 612 – 45; A.S.A. Z11.50—1945)

Test for Carbonizable Substances in White Mineral Oil (Liquid Petrolatum) (A.S.T.M. D 565 - 45; A.S.A. Z11.49-1945)

Test for Distillation of Gasoline, Naphtha Kerosine, and Similar Petroleum Products (A.S.T.M. D 86 - 45; A.S.A. Z11.10—1945)

Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. D 92 - 45; A.S.A. Z11.6—1945)

Test for Saponification Number of Petroleum Products by Color-indicator Titration (A.S.T.M. D 94 - 45; A.S.A. Z11.20—1945)

Test for Knock Characteristics of Motor Fuels (A.S.T.M. D 357 - 45; A.S.A. Z11.37—1945)

Sectional Committee C59:

Methods of Testing Shellac Used for Electrical Insulation (A.S.T.M. D 411 - 44; A.S.A. C59.18—1945)

Standard for Vulcanized Fiber (N.E.M.A. 43-87; A.S.A. C59.20—1945)

The following standards under the jurisdiction of sectional committees have been reaffirmed by the A.S.A. as follows:

Sectional Committee C59:

Specifications for Rubber Matting for Use Around Electric Apparatus or Circuits not Exceeding 3000 Volts to Ground (A.S.T.M. D 178 - 24; A.S.A. C59.4—1935; Reaffirmed 1945)

Specifications for Rubber Insulating Tape (A.S.T.M. D 119 - 38; A.S.A. C59.6—1939; Reaffirmed 1945)

Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators; (A.S.T.M. D 392 - 38; A.S.A. C59.10—1941; Reaffirmed 1945)

Sectional Committee H4:

Specifications for Hard-Drawn Copper Wire (A.S.T.M. B 1 - 40; A.S.A. H4.2—1941; Reaffirmed 1945)

Specifications for Medium-Hard-Drawn Copper Wire (A.S.T.M. B 2 - 40; A.S.A. H4.3—1941; Reaffirmed 1945)

Specifications for Bronze Trolley Wire (A.S.T.M. B 9 - 39; A.S.A. H4.5—1940; Reaffirmed 1945)

Specifications for Copper Trolley Wire (A.S.T.M. B 47 - 39; A.S.A. H4.6—1940; Reaffirmed 1945)

Under the Proprietary Standards Procedure, revisions of the following specifications were approved by the A.S.A.:

Committee B-5 on Copper and Copper Alloys, Case and Wrought:

Specifications for Free-Cutting Brass Rod and Bar for Use in Screw Machines (A.S.T.M. B 16 - 45; A.S.A. H8.1—1945)

Specifications for Copper Water Tube (A.S.T.M. B 88 - 45; A.S.A. H23.1—1945)

Specifications for Copper-Base Alloy Forging Rods, Bars and Shapes (A.S.T.M. B 124 - 45; A.S.A. H7.1—1945)

Committee D-1 on Paint, Varnish, Lacquer, and Related Products:

Methods of Test for Coarse Particles in Pigments, Pastes and Paints (A.S.T.M. D 185 - 45; A.S.A. K42.1—1945)

Committee D-11 on Rubber and Rubber-Like Materials:

Method of Test for Accelerated Aging of Vulcanized Rubber by the Oven Test Method (A.S.T.M. D 573 - 45; A.S.A. J5.1—1945)

Discharge of Sectional Committee:

During the year, Sectional Committee G8 on Zinc Coating of Iron and Steel has been discharged. For a number of years all of the work on standards relating to zinc coatings of iron and steel has been carried out in A.S.T.M. Committee A-5 on Corrosion of Iron and Steel which standards were subsequently transmitted to Committee G8 for approval. Since Committee A-5 represents a balanced membership, according to all A.S.A. requirements, it was the feeling in the sectional committee that full responsibility for acting upon standards, including their reference to the A.S.A., should rest with Committee A-5 and that the sectional committee be disbanded. After discussing this question with the organizations represented on Sectional Committee G8, the recommendation of the committee that it be discharged was approved by the Administrative Committee on Standards and concurred in by the A.S.A. with the understanding that any organizations represented on Committee G8 should be given the privilege of representation on Committee A-5.

United Nations Standards Coordinating Committee:

Several projects under consideration by the U.N.S.C.C. are of interest to the Society.

Standardization of Grades of Shellac and Methods of Testing.—A committee is being set up under the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products to handle international discussions on shellac. The project was suggested by the British Standards Institute.

Definition of Terms Relating to Heat Treatment of Metals.—A project for the unification of terms and definitions of terms relating to heat treatment of iron and steel was proposed by the British Standards Institute. According to the American viewpoint developed by the A.S.T.M., it was felt any discussions should relate to metals in general and not solely to iron and steel. It is proposed that any international discussions be handled by the present Joint Committee on Heat Treatment Definitions, expanding somewhat the personnel so to include representation from the Federal Government.

Methods of Testing Textiles.—The project on the unification of methods of testing textiles was proposed by the National Bureau of Standards. A special committee functioning under the Sectional Committee on Fastness of Colored Textiles (L14) has submitted a list of tests that might be used as a basis for these international discussions.

Respectfully submitted on behalf of the committee,

N. L. MOCHEL,
Chairman.

R. E. HESS,
Secretary.

APPENDIX A

RECOMMENDATIONS APPROVED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS AT ITS MEETING, AUGUST 27, 1945

TENTATIVES

Specifications for:

Electrodeposited Coatings of Lead on Steel (B 200 - 45 T), submitted by Committee B-8

Chromate Finishes on Electrodeposited Zinc, Hot-Dipped Galvanized, and Zinc Die-Cast Surfaces (B 201 - 45 T), submitted by Committee B-8

Metal Powder Sintered Bearings (Oil Impregnated) (B 202 - 45 T), submitted by Committee B-9

Methods of:

Compression Test for Shipping Containers (D 642 - 45 T), submitted by Committee D-10

Drop Test for Shipping Containers (D 775 - 45 T), submitted by Committee D-10

Chemical Analysis of Soaps Containing Synthetic Detergents (D 820 - 45 T), submitted by Committee D-10

Evaluating Degree of Resistance of Traffic Paint to Abrasion, Erosion, or a Combination of Both, in Road Service Tests (D 821 - 45 T), submitted by Committee D-1

Test for Total Aluminum and Aluminum Ion in Industrial Waters (D 857 - 45 T), submitted by Committee D-19 on Water for Industrial Uses

Manganese in Industrial Waters (D 858 - 45 T), submitted by Committee D-19

Test for Silica in Industrial Waters (D 859 - 45 T), submitted by Committee D-19

Chemical Analysis for Sulfur by Direct Combustion and Nitrogen in Steel (E 30 - 45 T), submitted by Committee E-3

Determination of Bismuth in Pig Lead (E 58 - 45 T), submitted by Committee E-3

Recommended Practice for:

Operating Light and Water Exposure Apparatus (Carbon-Arc Type) for Testing Paint, Varnish, Lacquer, and Related Products (D 822 - 45 T), submitted by Committee D-1

Sampling Boiler Water from Stationary Boilers (D 860 - 45 T), submitted by Committee D-19

REVISIONS OF TENTATIVES

Specifications for:

Electrodeposited Coatings of Nickel and Chromium on Steel (A 166 - 45 T), submitted by Committee B-8 on Electrodeposited Metallic Coatings

Electrodeposited Coatings of Nickel and Chromium on Zinc and Zinc-Base Alloys (B 142 – 45 T), submitted by Committee B-8

Asphalt Roofing Surfaced with Coarse Mineral Granules (D 249 – 45 T), submitted by Committee D-8

Methods of:

Test for Local Thickness of Electrodeposited Coatings (A 219 – 45 T), submitted by Committees A-5 and B-8

Test for Phthalic Anhydride Content of Alkyd Resins and Resin Solutions (D 563 – 45 T), submitted by Committee D-1.

Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products (D 609 – 45 T), submitted by Committee D-1

Recommended Practice:

For Chromium Plating on Steel for Engineering Use (B 177 – 45 T), submitted by Committee B-8

TENTATIVE REVISIONS OF STANDARDS

Specifications for:

Lampblack (D 209 – 41), submitted by Committee D-1

Bone Black (D 210 – 41), submitted by Committee D-1

Pure Chrome Green (D 212 – 41), submitted by Committee D 1

Prussian Blue (D 261 – 41), submitted by Committee D-1

Ultramarine Blue (D 262 – 41), submitted by Committee D-1

Chrome Oxide Green (D 263 – 41), submitted by Committee D-1

Titanium Dioxide Pigments (D 476 – 41), submitted by Committee D-1

Carbon Black (D 561 – 41), submitted by Committee D-1

Method of:

Chemical Analysis of White Pigments (D 34 – 39), submitted by Committee D-1

Test for Consistency of Exterior House Paints and Enamel-Type Paints (D 562 – 44), submitted by Committee D-1

WITHDRAWAL OF STANDARDS AND TENTATIVES

Method of:

Compression Testing of Corrugated and Solid Fiber Boxes (D 642 – 43), submitted by Committee D-10

Drop Test for Shipping Containers (D 775 – 44 T), submitted by Committee D-10

REVISION OF EMERGENCY ALTERNATE

EA – D 249c Tentative Specifications for Asphalt Roofing Surfaced with Coarse Mineral Granules (D 249 – 44 T), submitted by Committee D-8

WITHDRAWAL OF EMERGENCY STANDARD

ES-31 Emergency Specifications for Electrodeposited Coatings of Lead on Steel.

APPENDIX B

RECOMMENDATIONS APPROVED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS JULY 1, 1945–MARCH 16, 1946 (EXCLUDING AUGUST 27, 1945)

TENTATIVES

Specifications for:

Foundry Pig Iron (A 43 – 45 T), submitted by Committee A-3 on Cast Iron

Seamless Chromium-Molybdenum Alloy-Steel Pipe for Service at High Temperatures (A 280 – 46 T), submitted by Committee A-1 on Steel

Mild- to Medium-Strength Carbon-Steel Castings for General Application (A 281 – 45 T), submitted by Committee A-1

High-Strength Steel Castings for Structural Purposes (A 282 – 45 T), submitted by Committee A-1

Copper-Nickel-Zinc Alloy Wire (B 206 – 46 T), submitted by Committee B-5

Nickel-Chromium-Iron Alloy Castings (35-15 Class) for High Temperature Service (B 207 – 46 T), submitted by Committee B-4

Portland-Blast Furnace Slag Cement (C 205 – 46 T), submitted by Committee C-1

GR-S Synthetic Rubber Sheath Compound for Electrical Insulated Cords and Cable (D 866 – 46 T), submitted by Committee D-11

Method of:

Test for Strength of Welded Lead Wires for Electronic Devices and Lamps (B 203 – 45 T), submitted by Committee B-4

Test for Surface Flaws in Tungsten Seal Rod and Wire (B 204 – 45 T), submitted by Committee B-4

Test for Diameter by Weighing of Fine Wire Used in Electronic Devices and Lamps (B 205 – 45 T), submitted by Committee B-4

Chemical Analysis of Portland Cement (C 114 - 46 T), submitted by Committee C-1

Test for Fineness of Portland Cement by the Air-Permeability Apparatus (C 204 - 46 T), submitted by Committee C-1

Test for Acid Number of Dark Rosin (D 465 - 45 T), submitted by Committee D-17

Sampling and Testing Pine Tars and Pine-Tar Oils (D 856 - 45 T), submitted by D-17

Test for Evaluating Treated Textiles for Permanence of Resistance to Microorganisms (D 862 - 45 T), submitted by Committee D-13

Designating the Flow Temperature of Thermoplastic Molding Materials (D 863 - 45 T) submitted by Committee D-20

Test for Coefficient of Cubical Thermal Expansion of Plastics (D 864 - 45 T), submitted by Committee D-20

Heat Aging of Vulcanized Natural or Synthetic Rubber by Test Tube Method (D 865 - 46 T), submitted by Committee D-11

Test for Shipping Containers in Revolving Hexagonal Drum (D 867 - 46 T), submitted by Committee D-10

Chemical Analysis of Copper (Electrolytic Determination of Copper) (E 53 - 46 T), submitted by Committee E-3

Chemical Analysis of Copper and Copper-Base Alloys (E 62 - 46 T), submitted by E-3

Definition of:

Terms Relating to Naval Stores and Related Products (D 804 - 45 T), submitted by Committee D-17

Recommended Practice for:

A Universal System of Yarn Numbering (D 861 - 45 T), submitted by Committee D-13

REVISIONS OF TENTATIVES

Specifications for:

Carbon Steel Seamless Pipe for High-Temperature Service (A 106 - 45 T), submitted by Committee A-1

Alloy-Steel Bolting Materials for High-Temperature Service (A 193 - 45 T), submitted by Committee A-1

Naval Brass Rods, Bars and Shapes (B 21 - 46 T), submitted by Committee B-5

Seamless Copper Tubes (B 75 - 46 T), submitted by Committee B-5

Copper-Nickel-Zinc and Copper-Nickel Alloy Sheet and Strip (B 122 - 46 T), submitted by Committee B-5

Copper Rods, Bars, and Shapes (B 133 - 46 T), submitted by Committee B-5

Miscellaneous Brass Tubes (B 135 - 46 T), submitted by Committee B-5

Phosphor Bronze Rods, Bars, and Shapes (B 139 - 46 T), submitted by Committee B-5

Aluminum Bronze Rods, Bars, and Shapes (B 150 - 46 T), submitted by Committee B-5

Copper-Nickel-Zinc Alloy Rod, Bar, and Wire (B 151 - 45 T), submitted by Committee B-5

Phosphor Bronze Wire (B 159 - 46 T), submitted by Committee B-5

Rope-Lay-Stranded Copper Conductors Having Bunch-Stranded Members, for Electrical Conductors (B 172 - 45 T), submitted by Committee B-1

Rope-Lay-Stranded Copper Conductors Having Concentric-Stranded Members, for Electrical Conductors (B 173 - 45 T), submitted by Committee B-1

Bunch-Stranded Copper Conductors for Electrical Conductors (B 174 - 45 T), submitted by Committee B-1

Round Nickel Wire for Lamps and Electronic Devices (B 175 - 45 T), submitted by Committee B-4

Chromium-Nickel-Iron Alloy Castings (25-12 Class) for High-Temperature Service (B 190 - 45 T), submitted by Committee B-4

Beryllium-Copper Alloy Strip (B 194 - 46 T), submitted by Committee B-5

Beryllium-Copper Alloy Rod and Bar (B 196 - 46 T), submitted by Committee B-5

Beryllium-Copper Alloy Wire (B 197 - 46 T), submitted by Committee B-5

Vitrified Clay Filter Block for Trickling Filters (C 159 - 45 T), submitted by Committee C-15

Spec. for Air-Entraining Portland Cement for Concrete Pavements (C 175 - 46 T), submitted by Committee C-1

Molds for Test Specimens of Molding Materials Used for Electrical Insulation (D 647 - 45 T), submitted by Committee D-20 but now under D-9 jurisdiction

Phenolic Molding Compounds (D 700 - 45 T), submitted jointly by Committees D-9 & D-20

Insulated Wire and Cable: Heat-Resisting Synthetic Rubber Compound (D 754 - 46 T), submitted by Committee D-11

Insulated Wire and Cable: Performance Synthetic Rubber Compound (D 755 - 46 T), submitted by Committee D-11

Cellular Rubber Products (D 798 - 46 T), submitted by Committee D-11

A.S.T.M. Thermometers (E 1 - 45 T), submitted by Committee E-1

Methods of:

End-Quench Test for Hardenability of Steel (A 255 - 45 T), submitted by Committee A-1

Test for Temper of Strip and Sheet Metals for Electronic Devices (Spring-Back Method) (B 155 – 45 T), submitted by Committee B-4

Testing Wire for Supports Used in Electronic Devices and Lamps (B 157 – 45 T), submitted by Committee B-4

Test for Density of Fine Wire and Ribbon for Electronic Devices (B 180 – 45 T), submitted by Committee B-4

Test for Resistivity of Copper and Copper-Alloy Electrical Conductors (B 193 – 45 T), submitted by Committee B-1

Test for Air Content of Portland-Cement Mortar (C 185 – 46 T), submitted by Committee C-1

Test for Changes in Properties of Rubber and Rubber-Like Materials in Liquids (D 471 – 46 T), submitted by Committee D-11

Testing Cellular Rubber Products (D 552 – 46 T), submitted by Committee D-11

Test for Fastness of Colored Textiles to Light (D 506 – 45 T), submitted by Committee D-13

Test for Resistance of Textile Fabrics and Yarns to Insect Pests (D 582 – 45 T), submitted by Committee D-13

Test for Deformation of Plastics Under Load (D 621 – 45 T), submitted by Committee D-20

Test for Resistance of Textile Materials to Microorganisms (D 684 – 45 T), submitted by Committee D-13

Testing and Tolerances for Rope (D 738 – 45 T), submitted by Committee D 13

Testing and Tolerances for Spun, Twisted, or Braided Products Made from Flax, Hemp, Ramie, or Mixtures Thereof (D 739 – 45 T), submitted by Committee D-13

Testing Plywood, Veneer, and Other Wood and Wood-Base Materials, (D 805 – 45 T), submitted by Committee D-7

Definition:

Terms Relating to Textile Materials (D 123 – 45 T), submitted by Committee D-13

TENTATIVE REVISIONS OF STANDARDS

Specifications for:

Carbon and Alloy-Steel Nuts for Bolts for High-Pressure and High-Temperature Service (A 194 – 40), submitted by Committee A-1

Copper Brazed Steel Tubing (A 254 – 44), submitted by Committee A-1

Copper Rods for Locomotive Staybolts (B 12 – 45), submitted by Committee B-5

Free-Cutting Brass Rod and Bar for Use in Screw Machines (B 16 – 45), submitted by Committee B-5

Copper Water Tube (B 88 – 45), submitted by Committee B-5

Copper-Silicon Alloy Rods, Bars, and Shapes (B 98 – 45), submitted by Committee B-5

Copper-Silicon Alloy Wire for General Purposes (B 99 – 45), submitted by Committee B-5

Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 45), submitted by Committee B-5

Brass Wire (B 134 – 45), submitted by Committee B-5

Manganese Bronze Rods, Bars, and Shapes (B 138 – 45), submitted by Committee B-5

Leaded Red Brass (Hardware Bronze) Rods, Bars and Shapes (B 140 – 45), submitted by Committee B-5

Insulated Wire and Cable: Heat-Resisting Rubber Compound (D 469 – 41)

Methods of:

Testing Wool Felt (D 461 – 45), submitted by Committee D-13

Testing and Tolerances for Single Jute Yarn (D 541 – 41), submitted by Committee D-13

WITHDRAWAL OF STANDARDS

Specifications for:

Foundry Pig Iron (A 43 – 24), submitted by Committee A-3

Alloy-Steel Bolting Material for High-Temperature Service (A 96 – 44), submitted by Committee A-1

Drawn or Rolled Alloy, 80 per cent Nickel, 20 per cent Chromium, for Electrical Heating Elements (B 82 – 41), submitted by Committee B-4

Drawn or Rolled Alloy, 60 per cent Nickel, 15 per cent Chromium, and Balance Iron, for Electrical Heating Elements (B 83 – 41), submitted by Committee B-4.

APPENDIX II

REPORT OF ADMINISTRATIVE COMMITTEE ON RESEARCH

The Administrative Committee on Research has been more or less inactive for the past two years during the period when most A.S.T.M. technical committees were devoting their principal activities to standardization work. The committee has interested itself, however, in the activities of the National Research Council and in such other projects as affect our work. Among the latter are included our cooperation, through Committee D-20 on Plastics, with the research on plastics at Massachusetts Institute of Technology, underwritten by the Plastics Materials Manufacturers' Association; the proposals in Congress for a National Research Foundation; and support to the Engineering Foundation on the project of the Weldability of Carbon-Manganese Steel.

Current Problems under Consideration:

Suggested Increase of Principal of Research Fund.—In view of our present solicitation of moneys for the immediately necessary A.S.T.M. Building Fund, the committee has postponed indefinitely the drive to increase research funds. The officers of our technical committees, however, are being reminded of their authority to raise funds for individual projects, subject to the Regulations Governing Technical Committees.

Communication to Technical Committees.—The Administrative Committee is addressing a communication to the officers of all technical committees requesting them to check the latest list of research projects (December, 1943) and advise of corrections and particularly of additions of new projects. The committee officers will also be asked to furnish a brief statement of the scope of all new projects and brief reports on progress made in existing projects since December, 1943. Finally, the committee officers will be requested to review the status of war research in the fields covered by their respective committees and to advise whether there are any research projects growing out of the war that the committees would like to see established.

Research Projects Review.—A revision will be prepared of our December, 1943, "Review of A.S.T.M. Research Projects on Properties of Materials and on Methods of Testing."[1] The committee is considering expanding this review, to include appropriate reference to other lists of organized research problems in the materials field that may be available, as for example in the National Research Council.

Miscellaneous Projects under Discussion.—(1) Proposed study of Vapor Barriers under the sponsorship of Committee C-16 on Thermal Insulating Materials; (2) proposed aid to Committee D-3 on Gaseous Fuels in obtaining support from the American Gas Assn. for certain research projects on gaseous fuels; (3) Joint Committee on Effect of Temperature on the Properties of Metals of research on effect of temperature on various non-ferrous alloys, for which plans have been made but no work carried on in recent years; and (4) Testing of Parts and Assemblies. Due to our interest in this field of work we are proposing as a constructive move a joint meeting of our committee with the Administrative Committee on Simulated Service Testing.

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[1] See ASTM Bulletin, December, 1943.

Important Problems Relating to Society Policy:

In order that this committee may have more time to discuss important problems, it is planned to discontinue short meetings at our general A.S.T.M. gatherings in favor of full-time, one or two-day, committee meetings at headquarters. The first of these will be held in October this year, since much of the data required will be gathered from the annual reports and from the replies to the above-mentioned communication to the technical committee chairmen. We are also studying such questions as the proposed National Research Foundation, the building up of our A.S.T.M. research fund, etc., in order that the committee may be prepared to render such assistance as it can to the Executive Committee.

Respectfully submitted on behalf of the committee,

ROBERT J. MOORE,
Chairman.

C. L. WARWICK,
Secretary.

APPENDIX III

REPORT OF ADMINISTRATIVE COMMITTEE ON PAPERS AND PUBLICATIONS

The publication activities of the Society have continued particularly heavy during the past year. In addition to the regular publications, a number of special publications have appeared or have been in work.

As a matter of record, the 1945 publications distributed to members are listed below:

1945 *Proceedings*, 1060 pages, 6200 copies. It is to be noted that these *Proceedings* for the first time include papers that were not presented at the Annual Meeting but were offered for publication in the ASTM BULLETIN and selected by the Papers Committee for inclusion in the *Proceedings*. Available in May, 1946.

1945 Supplements to Book of A.S.T.M. Standards:

Part I, 419 pages, 8900 copies.
Part II, 245 pages, 7200 copies.
Part III, 525 pages, 7100 copies.

Index to 1945 Standards and Tentative Standards, 235 pages, 19,000 copies.

Year Book, 474 pages, 4750 copies.

ASTM BULLETIN, six issues, 492 pages, average edition, 8000 copies.

A statement of the volume of the principal publications for the past four years follows:

Publications	1942, pages	1943, pages	1944, pages	1945, pages
Proceedings	1225	1357	1139	1060
Book of A.S.T.M. Standards	4906	....	6042	....
Supplements to Book of A.S.T.M. Standards	....	1124	....	1189
A.S.T.M. Methods of Chemical Analysis of Metals	....	331	32	....
Index to A.S.T.M. Standards and Tentative Standards	206	218	218	235
Year Book	352	374	408	474
Spring Meeting Symposiums	....	135	140	....
	6689	3539	7979	2958

Special compilations of standards published from June 1, 1945 to April 15, 1946 are as follows:

A.S.T.M. Standards on Concrete and Concrete Aggregates (Committee C-9), 51 standards, 150 pages, 1000 copies.

A.S.T.M. Standards on Paint, Varnish, Lacquer, and Related Products (Committee D-1), 161 standards, 542 pages, 1500 copies.

A.S.T.M. Standards on Petroleum Products and Lubricants (Committee D-2), 101 standards, 556 pages, 5800 copies.

A.S.T.M. Standards on Electrical Insulating Materials (Committee D-9), 79 standards, 558 pages, 1100 copies.

A.S.T.M. Standards on Soaps and Other Detergents (Committee D-12), 29 standards, 192 pages, 1100 copies.

A.S.T.M. Standards on Plastics (Committee D-20), 109 standards, 554 pages, 4000 copies.

Special publications also issued:

Report of Committee A-5 on Corrosion of Iron and Steel, 21 pages, 500 copies.

Significance of Tests of Petroleum Products, 82 pages, 1500 copies.

Symposium on Analytical Colorimetry and Photometry, 76 pages, 2000 copies.

Round Table Discussion on Organizing the Classification of Industrial Waters, 48 pages, 1000 copies.

Symposium on Stress-Corrosion Cracking of Metals, 503 pages, 3000 copies. This is a joint publication with the American Institute of Mining and Metallurgical Engineers.

Symposium on Magnetic Particle Testing and Inspection, 128 pages, 1500 copies.

Final Report on Behavior of Ferritic Steels at Low Temperatures, by H. W. Gillett and F. T. McGuire, 218 pages (published in two parts), 1000 copies. This represents publication of data compiled for the War Metallurgy Committee of the National Academy of Sciences and the National Research Council.

Hardenability Chart, 53,000 copies.
Viscosity Temperature Charts, 22,500 copies.

CURRENT ACTIVITIES

Program for the Annual Meeting:

What is probably the heaviest program sponsored by the Society is contemplated for the 1946 Annual Meeting. This is due in part to the release of material that has been withheld during the war period, some of it by the Office of Scientific Research and Development, and to special solicitations that have been made by the Committee on Papers and Publications. It is also the reflection of some of the newer activities of the Society; for example the proposed Sessions on Fatigue, Parts and Assemblies, and Testing of Bearings are sponsored by the new Administrative Committee on Simulated Service Testing. An unusual number of symposia are being held. In addition to those mentioned above, the following are in prospect:

Symposium on pH Measurement—under the auspices of Committee E-1 on Methods of Testing.

Symposium on Spectroscopic Light Sources—under the auspices of Committee E-2 on Spectrographic Analysis.

Symposium on Atmospheric Weathering of Corrosion-Resistant Steel—under the auspices of Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys.

Symposium on Oil Procurement Practices—under the auspices of Committee D-2 on Petroleum Products and Lubricants.

Symposium on Freezing-and-Thawing Tests on Concrete—under the auspices of Committee C-9 on Concrete and Concrete Aggregates.

Special Sessions sponsored by the Joint Research Committee on Effect of Temperature on the Properties of Metals.

This may result in the withholding, for want of appropriate technical sessions, of presentation of some of the individual offers of papers. These papers, however, will be accepted for publication even though not presented at the meeting.

1946 Book of Standards:

The largest single publication in prospect during the year is the 1946 Book of A.S.T.M. Standards. The Book of Standards has now reached such a magnitude that some other provision will need to be made in order to keep down the size of the individual volumes. Consideration was given to the use of thinner paper, but the present indications are that such thin paper will not be available except the highest qualities at prohibitive cost. In lieu of this, consideration will need to be given to splitting the books into five volumes rather than the present three, but retaining the present Parts I, II, and III division, particularly since our present records with respect to the wishes of the individual members for the receipt of the Book of Standards are based upon the present Parts I, II, and III subdivision. The standards of Part I may thus be issued as Part Ia and Ib and those of Part III as Part IIIa and IIIb. The committee is now engaged in a study as to how the division can best be effected.

Compilations of Standards:

The list of compilations continues to grow and several new ones are in prospect, namely, on Light Metals and Alloys, Committee B-7; Glass, Committee C-14; and Water for Industrial Uses, Committee D-19.

Special Publications:

The number of special publications continues to increase. These frequently arise as a result of technical programs sponsored by the Districts of the Society. It is interesting, however, that a number of special publications become more or less permanent publications in that we are asked to publish supplements or revisions of earlier printings. This is the case with the Bibliography and Abstracts on Electrical Contacts which has an annual supplement. The Index to the

Literature on Spectrochemical Analysis has supplements which are printed periodically; an extensive supplement is to appear this year which will have an innovation of including brief abstracts. The X-Ray Diffraction Data Cards are added to annually. Special mention might be made of the Manual on Presentation of Data which was in considerable demand during the war years as the basic work in connection with quality control. The Significance of Tests of Petroleum Products is now in its fourth printing.

Special publications that are expected in due time are:

Symposium on Adhesives—sponsored by Committee D-14 on Adhesives at a meeting held in Atlantic City in October, 1945.

Symposium on Ultra High Voltage and High-Speed Radiography—sponsored by Committee E-7 on Radiography and held in conjunction with the Fifth Annual Convention of The American Industrial Radium and X-Ray Society, Inc., in Cleveland, February, 1946.

Symposium on Effects of Low Temperatures on Materials—sponsored by the Philadelphia District Committee at a meeting held in Philadelphia, March, 1946.

Symposium on Atmospheric Exposure Tests on Non-Ferrous Metals—sponsored by Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys and held during the Spring Meeting, February, 1946.

Discussion on Statistical Quality Control in Its Application to Specification Requirements—held during the Spring Meeting, February, 1946. The two papers appear in the March issue of the ASTM BULLETIN, and will also be published separately with discussion.

Some of the symposia at the annual meeting may result in special compilations, for example, the papers on Effect of Temperature on the Properties of Metals and on the Testing of Bearings. Published in this way, these papers may not appear in the *Proceedings*.

Punching Separate Standards for Loose Unit Binders:

In accordance with the recommendations of the Executive Committee all of the standards that have been printed for the past year have been punched to fit any standard three-ring binder. At the present time most of the standards have been punched. There has also been made available a three-post expansion type binder for sale to the membership, which comes in both 1- and $1\frac{1}{2}$-in. size. Both binders are bound in imitation leather and stamped in gold "Selected A.S.T.M. Standards."

FUTURE ACTIVITIES

Plans are now under way for the 1947 Spring Meeting of the Society for which a technical program on paints is contemplated sponsored by Committee D-1 on Paint, Varnish, Lacquer, and Related Products. Aside from this, it is expected that a number of papers will be submitted for publication during the year; some of these include reports as they are released covering activities during the war period. For example, several papers are expected dealing with plastic deformation of metals. This is a reflection of the newer policies with respect to publication, namely, that papers may be published throughout the year and not necessarily held for an annual meeting of the Society.

In connection with this general growth of our publications, mention should be made of the increasing use of the ASTM BULLETIN. Many papers presented at committee meetings are submitted for BULLETIN publication, and the BULLETIN is also used to some extent as a medium for preprinting papers prior to their inclusion in the *Proceedings*.

Respectfully submitted on behalf of the committee,

C. L. WARWICK,
Chairman.

APPENDIX IV

REPORT OF ADMINISTRATIVE COMMITTEE ON ULTIMATE CONSUMER GOODS

The steps leading to the formation of the Administrative Committee on Ultimate Consumer Goods will first be reviewed so that members of the Society may realize the very thorough and full deliberations which preceded the official organization of the committee.

Brief Résume of the Formation of the Committee:

In December, 1943, the Executive Committee appointed a Study Committee of twelve members whose duty it was to determine whether the Society, through its prevailing organization and functioning, was geared to meet war and post-war conditions, and to recommend such changes as would put the Society in a position to render the best possible service to its members, industry, and the nation at large.

This committee gave early consideration to one matter of long standing and of recurring nature, namely, as to how far the Society should go in the formulation of standards for ultimate consumer goods. In recognition of the import of the increasing activity in this field outside of the Society and of the work already begun by some of the standing committees within the Society, the Study Committee recommended the appointment of a special committee to make a thorough study of the manner and extent of Society participation in the development of definitions, test methods, and specifications for ultimate consumer goods. The recommendation pointed out that duplication of the activities of existing competent organizations was undesirable, and that certain specific exclusions such as foods, drugs, and cosmetics were advisable.

The Executive Committee approved of this recommendation in October, 1944, and a Special Committee on Ultimate Consumer Goods, composed of nine members under the chairmanship of A. W. Carpenter, was appointed. The Special Committee devoted two full days to a complete examination and discussion of all phases of its assigned problem, and submitted to the Executive Committee in January, 1945, a 7-page report summarizing its deliberations and containing nine definite recommendations. The essence of two of these recommendations was (1) that the Society should continue and conservatively extend its activities in the development of standards for ultimate consumer goods, with certain limitations, and (2) that an Administrative Committee on Ultimate Consumer Goods should be established with such authority and responsibility as the Executive Committee might delegate to plan and direct work by the committees of the Society in the field of ultimate consumer goods.

The Executive Committee discussed the report and each recommendation thoroughly, suggesting several minor modifications. It then proceeded to accept the report; in the same vote, it authorized the establishment of the Administrative Committee on Ultimate Consumer Goods and empowered the

President to appoint the chairman and members.[1]

The following scope was assigned to the committee:

The committee has for its functions the supervision of the Society's activities in the development of standards for ultimate consumer goods that permit of definitions, test data, or test limitations that can be measured by engineering methods, but not including assemblies except where evaluation of materials or workmanship is concerned.

Meetings:

Two day-long meetings of the Administrative Committee on Ultimate Consumer Goods have been held with complete attendance in each case. The first meeting was in Philadelphia on April 11, 1945; the second in New York City on September 12, 1945.

Definition of Ultimate Consumer Goods:

All deliberations of the Administrative Committee are predicated on the following definition of ultimate consumer goods prepared by the Special Committee referred to above:

> *Ultimate Consumer Goods* are materials or products which in the "as is" condition are intended for sale to an individual purchaser for his personal property or use, and not for fabrication for resale. Such an individual is the Ultimate Consumer.

General Policy re Development of Standards for Ultimate Consumer Goods:

The Administrative Committee has decided that the Society, in its work in the field of ultimate consumer goods, can best serve industry and the consumer by giving primary emphasis to the development of methods of test for use values of ultimate consumer goods. The development of specifications for such goods will be undertaken only when it appears that adequate methods of evaluation are available and that there is a demonstrated need for such specifications.

Suggested Form of Standards for Ultimate Consumer Goods:

The Administrative Committee has given considerable time and study to the form and content which standards pertaining to ultimate consumer goods should have. It has prepared a Method of Test Form and a Specification Form in which are embodied the recommendations on this subject made by the Special Committee, together with some ideas of its own. Attention is directed to some of the features wherein these suggested Forms differ from the usual A.S.T.M. standard:

Title.—Specific and naming end-use if possible; includes phrase "applicable to ultimate consumer goods"; serial designation preceded by CG.

Special Clauses in Preamble.—The first, disclaiming responsibility of A.S.T.M. for use of standard in labeling, certification, and advertising; the second, warning the user of the standard that the data upon which it is based may not adequately cover all use-values.

Scope.—Emphasis upon the clear, precise, and definite statement of the applicability and limitations of the method of test or specification; end-uses explicitly stated.

Definitions or Descriptions of Terms.—Recommends use of simple consumer terminology; use of English units of measurement in place of metric.

Ultimate Consumer Acceptance.—What constitutes acceptance, and recourse of consumer in event of unsatisfied claims.

In order to clarify still further the conception of what a standard for ultimate consumer goods should look like, the committee has selected two existing

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[1] For a fuller statement, including announcement of the membership of this committee, see Annual Report of the Executive Committee, *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 21 (1945). See also ASTM BULLETIN, March, 1945, p. 5.

A.S.T.M. standards, which are being revamped into the form and content they might have if designed for ultimate consumer use. When these are ready, it is planned to send copies to the chairmen of the committees concerned for their information and comments.

Communication to Chairmen of Standing Committees:

A communication has recently been sent to the chairmen of standing committees directing their thought to the possibility of activity in the ultimate consumer goods field, informing them of the functions of the Administrative Committee, of the general policy adopted by it, and requesting their comments upon the suggested forms. It is too early to report results at this time.

Research Program on Methods of Determining Consumer Wants:

It would seem pertinent to record in this report the status of the research program because of the direct application it will have to the work of the Administrative Committee. It will be recalled that the Executive Committee accepted a recommendation of the Special Committee that "the Executive Committee shall consider the establishment of a research project within A.S.T.M. for the development of techniques for obtaining knowledge of consumer wants and for accumulating data on which to base specifications and methods of test for ultimate consumer goods. It is contemplated that this project should utilize social, statistical, psychological, engineering, and economic approaches." Since that time the National Research Council and the Social Science Research Council have formed a joint committee to work on methodological problems in the measurement of consumer wants, public opinion, and social attitudes. C. L. Warwick is the A.S.T.M. representative.

Despite the organization of the joint NRC-SSRC committee, the Administrative Committee has formally voted in favor of setting up a research committee within the A.S.T.M. in the belief that it would cover many problems in the consumer field outside the scope of the joint committee. The Administrative Committee has no recommendation to make at this time, however. When the program of the joint committee is available, the Administrative Committee will be able to recommend a program which will avoid duplication of effort.

Standing Committee Organization and Procedure in Developing Standards for Consumer Goods:

The question of how work on standards for ultimate consumer goods might be handled within a standing committee poses a problem which has been given considerable attention by the Administrative Committee. The question arises because of the desirability of having a reasonable balance of interests between producers and ultimate consumers when work on consumer goods standards is undertaken. Those now classified as consumers on standing committees are very generally representative of the industrial consumers, and may or may not be qualified to serve as representatives of the ultimate consumer. In any event, the Administrative Committee has agreed that A.S.T.M. standards on consumer goods will not carry proper weight if in their establishment there has not been competent representation from such groups as the national women's clubs, American Home Economics Assn., departments of home economics in the colleges and government bureaus, consumer cooperatives, labor organizations, retail, mail order and department stores, magazine publishers, etc.

Must such groups take out membership in the A.S.T.M. in order to participate in the work? Since most standing committees as at present constituted have only a small ultimate consumer representation, if any, should a sectional committee, with suitable balance of interests, be set up within or without the framework of the standing committee to develop the standards? Must approval of the standards for submittal to the Society be by vote of the entire standing committee or will approval by the sectional committee alone be recognized? What changes in the Regulations Governing Technical Committees may be needed to accomplish this or equivalent procedure? The Administrative Committee has not evolved a definite plan at present, but presents this material as one of the current problems under consideration.

Other Plans:

The Administrative Committee is planning to approach a group of organizations which have been associated with consumer research to learn what demands there are for standardization work in the field of ultimate consumer goods. It also expects to inquire of the officers of technical committees on what items of consumer interest they are working or on which work should be undertaken. Information from these two sources is expected to be helpful in giving direction to the committee's future activities.

The committee is considering the possibility of preparing for the guidance of technical committee chairmen a suggested procedure in the development of test methods for ultimate consumer goods.

Respectfully submitted on behalf of the committee,

HERBERT J. BALL,
Chairman.

R. E. HESS,
Secretary.

APPENDIX V

REPORT OF ADMINISTRATIVE COMMITTEE ON SIMULATED SERVICE TESTING

Following preliminary discussions and meetings held late in 1945 and early in 1946, the Executive Committee of the Society authorized the formation of the Administrative Committee on Simulated Service Testing,[1] with the following assigned scope:

> This committee has for its functions the supervision of the development and standardization of methods of test of simple or composite materials in actual or simulated service conditions and environment, in so far as performance has a bearing on the properties of the material. It is understood that this may involve the testing of processed parts under such conditions.

The committee held its initial meeting on April 18, 1945, and proceeded to define the scope and activities of the committee, establish its organization, personnel, etc. The agenda of this meeting called for a careful survey of all pertinent suggestions for committee action, and it was decided to consider the activities of the Society in fatigue testing as the prime item for action.

The committee has thus far accomplished the following:

1. It has recommended the reorganization of fatigue testing activities in the Society by establishing a new Committee E-9 on Fatigue, suggesting the organization, personnel, scope, etc. The scope has been assigned as follows:

> To formulate methods for the determination of fatigue characteristics of simple and composite materials, components, and processed parts; to promote research in these fields; and to coordinate such Society activities conducted by other technical committees.

The present Research Committee on Fatigue of Metals will be discontinued as such and its activities transferred to the new committee as a subcommittee on research.

These arrangements have been approved by the Executive Committee and the new committee will be organized during the 1946 Annual Meeting.

2. It has recommended the formation of a new Committee E-6 on Methods of Testing Building Constructions, including suggested scope, organization, initial personnel, etc. The scope of the committee is:

> To formulate methods of test for building constructions, including elements, connections and assemblies, under actual or simulated service conditions, applicable to the evaluation of such factors as materials, design, construction and fabrication.

This committee held its organization meeting in Washington, D.C., on January 23, 1946.[2]

3. It has initiated and, with the aid of the Administrative Committee on Papers and Publications, has developed Symposiums on Fatigue and on the Testing of Parts and Assemblies (the latter jointly with the Society for Experimental Stress Analysis) that will be a part of the 1946 Annual Meeting. The committee has also arranged for a series of papers on the Testing of Bearings, concerning

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[1] See Annual Report of Executive Committee *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 21 (1945).

[2] For news account of this meeting, see March, 1946 ASTM BULLETIN, p. 49.

which there have been a number of important developments in recent years. These papers constitute a further contribution to the discussion of methods of test of an important type of assembly.

Excellent progress has been made in the development of the programs for all three of these topics, which should serve effectively to place the desired emphasis upon this subject in A.S.T.M.

4. It has taken initial steps in the proper allocation within the Society of work on "sandwich-type" materials and will participate in a conference on this subject on June 7.

Problems of a current nature now before the committee include the following:

1. Continuation of (4) above.

2. Consideration of already suggested testing in the fields of (*a*) aircraft equipment, (*b*) automotive equipment, and (*c*) ordnance equipment.

During the process of organization of the committee, suggestions were requested for activities in this field which resulted in a rather lengthy list of meritorious suggestions. The above items of current activity are among the most important of those suggested, and the future work of the Administrative Committee will involve the pursuit of all of the current activities and suggestions now on hand (unless superseded by ones more important) as rapidly as possible.

Respectfully submitted on behalf of the committee,

L. L. WYMAN,
Chairman.

C. S. COLE,
Secretary.

APPENDIX VI

REPORT OF ADMINISTRATIVE COMMITTEE ON DISTRICT ACTIVITIES

The following constitutes a report to the Executive Committee respecting status of the work now before the Administrative Committee on District Activities.

Upon the presentation of its first report on October 10, 1945, the Executive Committee approved the recommendation that the simple designation "District" be used to refer to those groups of members residing within certain rather loosely defined geographic areas. In that report it was indicated that the present Charter for A.S.T.M. Districts and the proposed District Manual would be rewritten and submitted for approval.

Following the adoption of the designation "District," both the Charter and the Manual were rewritten and after thorough study by the committee, are submitted at this time for your approval.[1] In recommending adoption by the Executive Committee, the Administrative Committee recognizes the importance that each of these instruments have the approval of the several Districts through their governing bodies. The Charter and the Manual are being distributed to the District officers with the recommendation that both be reviewed and discussed at a meeting of the District officers to be held during the 1946 Annual Meeting. The Administrative Committee recommends that the Charter become effective as of January 1, 1947.

The Administrative Committee approved the organization of a new District in the New England states and recommends that the organization of this District be consummated on the present basis as soon as possible. The exact name of the District should be a decision of its governing body.

Concerning the affiliation of A.S.T.M. Districts with local councils of technical or engineering societies, the Administrative Committee believes that such affiliations should be decided upon the merits of each individual case, and that the committee should be consulted and asked to review any proposal for affiliation which should originate in the respective Districts.

A study is under way in the committee that concerns the more exact definition of the areas comprising each District. An objective in that study is a plan that will fit into the ultimate growth of the Society and associate, ultimately, all members with some District.

At present there are ten organized Districts; a new one in the New England States is approved, and Districts that may be designated as the Washington District and the Cincinnati District are under consideration.

In order to stimulate interest in the District idea, the committee has proposed a meeting of all District officers on June 26, 1946.

The Administrative Committee reiterates its belief in the development of Districts as a stimulus to Society growth and value to industry generally.

Respectfully submitted on behalf of the committee,

C. H. Fellows,
Chairman.

R. J. Painter,
Secretary

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[1] The Charter and Manual are not included with this printing. They have been referred to the Administrative Committee for consideration of some proposed modifications.—Ed.

REPORT OF COMMITTEE A-1[*]

ON

STEEL

In the transition from wartime to peacetime operations there have been some serious and perplexing problems confronting the steel industry and also those who use the many diverse steels and steel products. Time has frequently been the essence in reaching satisfactory conclusions.

An almost exactly analogous situation has faced Committee A-1 on Steel. The scheduling and producing of rolling, forging, and casting operations frequently involves many weeks and months, and consequently even if Committee A-1 could have acted over-night in removing the large number of emergency alternate provisions and emergency specifications which were issued as wartime measures, no advantage would have accrued. In fact, there would have been much confusion. Now, however, as this report shows, the large number of widely used specifications are for the most part back on a peacetime footing, with many of the wartime requirements that have proved to be worth while and constructive retained as part of the regular specifications.

Meetings.—Although there had not been any meetings of the main Committee A-1 on Steel from June, 1944, when a series of meetings was held in New York, N. Y., June 26 to 28, 1944, until the Pittsburgh meetings, February 25 to 27, 1946, there have been in the interim many subcommittee meetings and a series of these was held in Philadelphia, Pa., November 5 and 6, 1945. These subcommittee meetings enabled Committee A-1 to take action on numerous recommendations since under the A.S.T.M. Regulations governing Standing Committees upon completion of subcommittee letter ballots these recommendations were subsequently referred to the main committee without the necessity of a meeting. Many such actions have been cleared through the Administrative Committee on Standards as noted later in this report. Meanwhile this situation which has prevented action on changes in formal standards has had the salutary effect of concentrating production with standards as they existed during the war period but modified by the emergency provisions. There will be a series of committee meetings, both subcommittee and main, during the 1946 Annual Meeting in Buffalo in June.

Personnel.—Since the 1944 Annual Meeting when the committee's last report[1] was submitted, there have been many changes in personnel. The number of voting members as of June, 1944 was 242, compared with the present figure, 258. Including additional representatives of companies and other organizations, there are now about 375 individuals participating in the committee's work. Of the total voting members, 138 are classified as nonproducers and 120 as producers; of the former, 99 are consumers and 39 are general interest members.

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[*] Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 44, p. 63 (1944).

Members Retiring.—During the war period a number of the long-time members of Committee A-1, who in the course of events as they advanced in years would doubtless have retired from their industrial work, have continued with their responsibilities, contributing much to the war effort and releasing other men for various responsibilities. By the time of the 1946 Annual Meeting, a number of men in this and related categories will have retired and Committee A-1 wishes to acknowledge its appreciation of their long-time activities in its behalf. A list of such men and the year they became members of Committee A-1 includes the following:

O. U. Cook, Tennessee Coal, Iron and Railroad Co., since 1921,
R. H. Dibble, Carnegie-Illinois Steel Corp., since 1926,
Chester Hacking, Wm. H. Haskell Manufacturing Co., since 1930,
Glen F. Jenks, Consulting Engineer, since 1934,
L. S. Marsh, Inland Steel Co., since 1922,
W. R. Shimer, Bethlehem Steel Co., Inc., since 1922 (deceased, Summer, 1946),
J. J. Shuman, Jones & Laughlin Steel Corp., since 1911, and
Victor Willoughby, American Car and Foundry Co., since 1924.

Losses by Death.—During the past two years Committee A-1 has lost by death a number of members who have participated actively in its work, including M. C. Blest, Pressed Steel Car Co., a member of the committee since 1923, H. N. Boetcher, representative of the Consolidated Gas, Electric Light and Power Co. of Baltimore, who had been active particularly in the work on power piping for many years, and Howard J. Kerr, Babcock & Wilcox Co., an authority on materials for boilers and pressure vessels and a keen observer of the advantages and weaknesses of various steels at elevated temperatures. Committee A-1 acknowledges its debt to these men and records its appreciation of their long-time and valuable services.

Honorary Membership.—While over the years Committee A-1 has tendered homage to many of its leading members and has, by election to official positions in the committee and subcommittees, acknowledged the great services of some of the men, there has been no provision in its regulations by which some of the signal activities of the various individuals could be recognized. Probably the most cherished honor that can come to an individual on Committee A-1 is the friendship and respect of other members for his activities and judgment. Nevertheless, the committee has instituted a new class of membership known as "Honorary" and a number of members who have rendered distinguished service to the committee have been selected for this distinction. Each of these men, an outstanding leader in his field, had for many years rendered notable service to the committee. Some of the men are still active in A-1, others have recently retired, and some have been in retirement for a number of years. The following list includes the name of the organization with which the individual was affiliated during the major years of work with the committee:

Allan W. Carpenter, New York Central System
O. U. Cook, Tennessee Coal, Iron and Railroad Co.
L. H. Fry, Edgewater Steel Co., now Director, The Steam Locomotive Research Institute
W. C. Hamilton, American Steel Foundries
J. O. Leech, Carnegie-Illinois Steel Corp.
L. S. Marsh, Inland Steel Co.
C. F. W. Rys,* Carnegie-Illinois Steel Corp.
J. J. Shuman, Jones & Laughlin Steel Corp.

*** Deceased, October, 1946.**

F. N. Speller, National Tube Co.
F. M. Waring, Pennsylvania Railroad Co.
C. D. Young, Pennsylvania Railroad Co.

EMERGENCY SPECIFICATIONS AND EMERGENCY ALTERNATE PROVISIONS

Appropriate articles and references have appeared in the Society publications including the ASTM BULLETIN with respect to the activities of Committee A-1 in the National Emergency Steel Specifications project, a War Production Board sponsored activity, and to the issuance as a result of this work of many emergency alternate provisions and the issuance of complete emergency specifications. A large portion of the personnel of the Technical Advisory Committees by means of which the NESS project functioned were A-1 members, and with one or two exceptions the TAC chairmen were active in Committee A-1 work.

Although the intensive work of the various men has been suitably acknowledged by the War Production Board, Committee A-1 wishes to record its appreciation for the great amount of time and effort expended by its members in solving the diverse problems that arose. Each can take pride in whatever his contributions may have been because unquestionably the emergency activities were of very great importance both in expediting the production of so many steel products and in conserving critical and strategic alloys.

In order that there shall be a permanent record of the emergency specifications and emergency alternate provisions affecting the A.S.T.M. steel specifications, that were issued as wartime measures, a list of the serial designations with the dates of issuance appears in Table I.

TABLE I.—LIST OF EMERGENCY STANDARDS AND EMERGENCY ALTERNATE PROVISIONS PREPARED BY COMMITTEE A-1.

Designations	Date of Issue and Revision
EMERGENCY STANDARDS	
Bearing Steel:[a]	
ES-5a	April 28, 1942, Revised Sept. 16, 1942
Heavy Forgings:[b]	
ES-21	Jan. 30, 1943
ES-22	Jan. 30, 1943
ES-23a	Jan. 30, 1943, Revised Dec. 11, 1944
ES-24	Jan. 30, 1943
ES-25	Jan. 30, 1943
ES-26	Jan. 30, 1943
ES-27	Jan. 30, 1943
EMERGENCY ALTERNATE PROVISIONS[c]	
Steel Plates for Pressure Vessels:	
EA-A 70a	Nov. 17, 1943, Revised Aug. 28, 1944
EA-A 89	Aug. 30, 1943
Concrete Reinforcement Steel:	
EA-A 160	April 6, 1942
Steel Rails and Accessories:	
EA-A1a	April 6, 1942, Amended Oct. 20, 1943
EA-A 183	April 6, 1942
Steel Billets, Forgings, and Axles:	
EA-A 21	Oct. 5, 1942
EA-A 235	Jan. 30, 1943
EA-A 236a	Aug. 12, 1942, Revised May 8, 1943
EA-A 237	Jan. 30, 1943
EA-A 238	Oct. 5, 1942
EA-A 274	Aug. 28, 1944
Steel Wheels and Tires:	
EA-A 25a	June 22, 1942, Revised Aug. 24, 1942
EA-A 26	April 28, 1942
EA-A 57	June 22, 1942
EA-A 186	June 22, 1942
EA-A 244	June 22,1942
Steel Castings:	
EA-A 27	Feb. 24, 1942
EA-A 87	Feb. 24, 1942
EA-A 95	April 26, 1944
EA-A 148a	Feb. 24, 1942, Revised April 27, 1943
EA-A 157	April 26, 1944
EA-A 215	Feb. 24, 1942
EA-A 216a	April 6, 1942, Revised April 26, 1944
EA-A 217	April 6, 1942
Pipe:	
EA-A 53	Jan. 30, 1943
EA-A 120	Aug. 24, 1942
EA-A 134	Aug. 18, 1942
EA-A 135	Aug. 24, 1942
EA-A 139a	Aug. 18, 1942 Revised April 8, 1943
EA-A 158a	June 1, 1943, Revised Aug. 28, 1944
EA-A 206	June 1, 1943
EA-A 211	Aug. 18, 1942
Tubes:	
EA-A 83	Aug. 18, 1942
EA-A 161	Aug. 18, 1942
EA-A 178	Aug. 18, 1942

TABLE I.—*Continued*

Designations	Date of Issue and Revision
EA-A 179	Aug. 18, 1942
EA-A 192	Aug. 18, 1942
EA-A 199	Aug. 18, 1942
EA-A 200	Aug. 18, 1942
EA-A 209	Aug. 18, 1942
EA-A 213a	Aug. 24, 1942, Revised Aug. 28, 1944
EA-A 214a	Aug. 18, 1942, Revised Aug. 30, 1943
EA-A 226	Aug. 18, 1942
EA-A 249a	Aug. 24, 1942, Revised Aug. 28, 1942
EA-A 250	Aug. 24, 1942
EA-A 268	Oct. 25, 1944
EA-A 269	Oct. 25, 1944
EA-A 270	Oct. 25, 1944
EA-A 271	Oct. 25, 1944
Steel Bolting Materials:	
EA-A 194	April 6, 1942

[a] This Emergency Standard was transferred to the status of tentative on June 27, 1946 and is now published under the A.S.T.M. Designation A 295 - 46 T in the 1946 Book of A.S.T.M. Standards, Part I-A, p. 980.

[b] These Emergency Standards were transferred to the status of tentative on July 10, 1946 and are now published respectively under the A.S.T.M. Designations A 288 to A 294, inclusive, and appear in the 1946 Book of A.S.T.M. Standards, Part I-A, pp. 837 to 862.

[c] All of these Emergency Alternate Provisions were withdrawn on June 27, 1946. Certain of these emergency alternate provisions have, however, resulted in revisions adopted this year in the standards and tentatives for steel and steel products.

For the most part, reasons for the emergency actions have been noted in various Society publications including the ASTM BULLETIN. In recommending the withdrawal of many of the provisions, Committee A-1 is retaining some of the requirements which may involve new alloys and test requirements. These are being embodied in the regular standards and tentatives. In the case of the complete emergency specifications, they have been transferred without revision from the status of emergency to that of tentative.

TENTATIVES AND REVISIONS OF TENTATIVES APPROVED DURING THE PAST TWO YEARS

Since June, 1944, when the last report of Committee A-1 was submitted to the Society, a number of recommendations of Committee A-1 were accepted by the

TABLE II.—RECOMMENDATIONS ACCEPTED BY ADMINISTRATIVE COMMITTEE ON STANDARDS, INCLUDING RECORD OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
New Tentative Specifications for:			
Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 - 44 T), *accepted October 25, 1944*.......	74	1	55
Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 - 44 T), *accepted October 25, 1944*.......	74	1	55
Seamless and Welded Austenitic Stainless Steel Tubing for the Dairy and Food Industry (A 270 - 44 T), *accepted October 25, 1944*..................	68	1	61
Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 - 44 T), *accepted October 25, 1944*	67	1	62
Carbon-Steel Blooms, Billets, and Slabs for Forgings (A 273 - 44 T), *accepted August 28, 1944*	111	2	70
Alloy-Steel Blooms, Billets, and Slabs for Forgings (A 274 - 44 T), *accepted August 28, 1944*	104	2	76
Seamless Chromium-Molybdenum Alloy-Steel Pipe for Service at High Temperatures (A 280 - 46 T), *accepted January 10, 1946*; revised June 27, 1946.....	114	1	88
Mild- to Medium-Strength Carbon-Steel Castings for General Application (A 281 - 45 T), *accepted November 14, 1945*.....................	121	4	87
High-Strength Steel Castings for Structural Purposes (A 282 - 45 T), *accepted November 14, 1945*.....................	119	3	80
New Tentative Methods of Test for:			
Magnetic Particle Testing and Inspection of Commercial Steel Castings (A 272 - 44 T), *accepted August 28, 1944*.......	114	1	67
Magnetic Particle Testing and Inspection of Heavy Steel Forgings (A 275 - 44 T), *accepted August 28, 1944*.........	114	2	66
Revisions of Tentative Specifications for:			
Seamless Carbon-Steel Pipe for High-Temperature Service (A 106 - 45 T), *accepted November 14, 1945*..................	103	0	99
Seamless Alloy-Steel Pipe for High-Temperature Service (A 158 - 44 T), *accepted August 28, 1944*....................	93	0	88
Alloy-Steel Bolting Materials for High-Temperature Service (A 193 - 45 T), *accepted November 14, 1945*..................	113	1	87
Carbon-Steel Seamless Drum Forgings (A 266 - 44 T), *accepted August 28, 1944*........	109	2	70
Revision of Tentative Method of Test for:			
End-Quench Test for Hardenability of Steel (A 255 - 45 T), *accepted November 14, 1945*....	129	0	73

TABLE II.—*Continued*

Items	Affirmative	Negative	Ballots Marked "Not Voting"
Tentative Revisions of Standard Specifications for:			
Steel for Bridges and Buildings (A 7 – 42), *accepted August 28, 1944*........	101	1	81
Mild Steel Plates (A 10 – 39) *accepted August 28, 1944*.....	101	1	81
Low Tensile Strength Carbon-Steel Plates of Structural Quality for Welding (A 78 – 43), *accepted August 28, 1944*...	101	1	81
Commercial Quality Hot-Rolled Bar Steels (A 107 – 42), *accepted August 28, 1944*........	120	5	57
Commercial Cold-Finished Bar Steels and Cold-Finished Shafting (A 108 – 36), *accepted August 28, 1944*..............	112	3	68
Electric-Fusion-Welded Steel Pipe (Sizes 8 in. to but not Including 30 in.) (A 139 – 42), *accepted August 28, 1944*......	82	0	99
Carbon- and Alloy-Steel Nuts for Bolts for High-Pressure and High-Temperature Service (A 194 – 40), *accepted November 14, 1945*....................	112	0	90
Carbon-Steel Forgings for General Industrial Use (A 235 – 42), *accepted August 28, 1944*...	109	2	70
Carbon-Steel Forgings for Locomotives and Cars (A 236 – 42), *accepted August 28, 1944*......	109	2	70
Alloy-Steel Forgings for General Industrial Use (A 237 – 42), *accepted August 28, 1944*.......	109	2	70
Alloy-Steel Forgings for Locomotives and Cars (A 238 – 42), *accepted August 28, 1944*......	109	2	70
Low-Alloy Structural Steel (A 242 – 42), *accepted August 28, 1944*..................	101	1	81
Carbon-Steel and Alloy-Steel Ring and Disk Forgings (A 243 – 43), *accepted August 28, 1944*..................	109	2	70
Copper Brazed Steel Tubing (A 254 – 44), *accepted November 14, 1945*....................	88	0	114
Withdrawal of Specifications for:			
Alloy-Steel Bolting Material for High-Temperature Service (A 96 – 44), *accepted November 14, 1945*....................	113	1	87
Carbon-Steel and Alloy-Steel Blooms, Billets and Slabs for Forgings (A 248 – 41 T), *accepted August 28, 1944*.........	107	0	75

Administrative Committee on Standards. The new and revised specifications are listed, with the date of approval, in Table II.

Table II does not include actions on emergency specifications or emergency alternate provisions which are noted elsewhere in this report.

COMMENTS ON RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

As announcement has been made from time to time in the ASTM BULLETIN of the new and revised specifications, there have also been given some notes or discussion on the background and reasons for the recommendations. Consequently, except for some significant groups of specifications which follow, those concerned are referred to the respective issues of the ASTM BULLETIN.

Stainless Steel Tubing:

While it had been evident for some time that there was a rather serious gap in the A.S.T.M. steel specifications with respect to so-called stainless steel tubing, attempts to draft specifications were somewhat fruitless because of conflicting opinions and other obstacles. However, through the work of the NESS project, which urged that specifications for these various products be made available, there was established a joint subcommittee of Committee A-1 and Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys, which included leading consumers and producers of the various classes of stainless tubing. In the meanwhile there had been developed by the American Iron and Steel Institute Technical Committee on Tubular Products proposed specifications, and as a result of these factors, agreement was finally reached on the four tentative specifications A 268, A 269, A 270 and A 271 noted in Table II. Because of war production problems, there were issued simultaneously with the new tentatives, emergency provisions which aided in expediting production and conserving critical elements. These emergency provisions governed during the war period.

Magnetic Particle Testing:

The already widespread and increasing use of magnetic particle methods for testing and inspecting various steel products, and the consequent confusion which was arising with respect to methods of magnetizing, type of powder or liquid to be used, and such factors, made it very desirable for the Society to consider undertaking some work in this field. Committee A-1, because of its concern with heavy forgings and with steel castings where the magnetic particle methods were very widely applied, agreed to undertake drafting recommended practices or test methods. Depending on the outcome of this work and the utility of the methods, further studies involving related methods such as fluorescent penetrants, etc., would be evaluated. As a result of the work of special sections organized particularly for this purpose, the Tentative Methods A 272 and A 275 noted in Table II were agreed on.

End Quench Test for Hardenability:

The revisions in the Tentative Method of End-Quench Test for Hardenability of Steel (A 255 – 45 T) essentially embody a revised chart for plotting the curves, which chart has a reduced scale compared with the earlier one and embodies other improvements. At the same time changes were made in the text of the method to bring the procedures more nearly in line with developments in the use of the test.[2]

Blooms, Billets and Slabs for Forgings:

The specifications for carbon-steel and alloy-steel blooms, billets and slabs for forgings, Specifications A 273 and A 274 respectively, brought up to date the requirements for these materials, and as a result of developments in the forging industry the committee was able to incorporate a number of special requirements which could become a part of the specifications upon agreement between the consumer and producer. Many of those purchasing material for reforging thus could use the standard specifications and meet certain special requirements needed for their particular products. No attempt was made to reduce the number of grades which might be employed but rather to make the specifications, both carbon and alloy, quite all-inclusive.

Pipe and Bolting for High-Temperature Service:

The sudden failure of a carbon-molybdenum grade of pipe in a high-temperature-high-pressure installation about two years ago caused a flurry of excitement, with much ensuing study and research to determine the causes. The problem was particularly pressing since there were many other installations of this particular grade of material which affords the necessary resistance to creep at elevated temperatures. The failure occurred in the weld-affected zone and was attributed to so-called graphitization or the formation of chain-like graphite nodules. Whether the use of aluminum in killing of the steel contributed to graphitization has been discussed in many technical papers since then, but the Committee's Subcommittee on Materials for High-Temperature Service was essentially concerned in developing or standardizing on a material which would inhibit graphitization. Since the incorporation of about 40 to 60 points of chromium seemed to provide the best safeguard, the new Specifications for Carbon-Molybdenum Steels (A 280 – 46 T) were agreed on after considerable discussion. Apparently no evidence exists to show that this material has graphitized below say 925 F., but only time and widespread usage will prove the worth of the material.

Meanwhile the studies continue of

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[2] See Editorial Note, p. 113.

TABLE III.—ANALYSIS OF LETTER BALLOT VOTE.

Items Specifications for the materials indicated	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVES			
Carbon-Steel Plates, Low and Intermediate Tensile Strengths of Structural Quality for Plates 2 in. and Under in Thickness (to replace Specs. A 10 and A 78)	108	0	79
Carbon-Silicon Steel Plates over 2 in. in Thickness of Intermediate Tensile Strength for Machine Parts and General Construction	108	0	79
Heat-Treated Alloy-Steel Bars	110	0	77
Rolled Carbon-Steel Locomotive Frames	79	2	114
Carbon-Chromium Ball and Roller-Bearing Steels (Tentative) (A 295 – 46 T)	83	0	104
II. REVISIONS OF TENTATIVES			
Seamless Carbon-Steel Pipe for High-Temperature Service (A 106 – 45 T)	96	15	75
Seamless Alloy-Steel Pipe for High-Temperature Service (A 158 – 44 T)	116	0	71
Alloy-Steel Bolting Materials for High-Temperature Service (A 193 – 45 T)	106	0	81
Seamless Carbon-Molybdenum Alloy-Steel Pipe for High-Temperature Service (A 206 – 44 T)	116	0	71
Alloy-Steel Castings Suitable for Fusion Welding for High-Temperature Service (A 217 – 44 T)	106	0	86
Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 44 T)	107	0	85
Seamless Chromium-Molybdenum Alloy-Steel Pipe for Service at High Temperatures (A 280 – 46 T)	116	0	71
Mild- to Medium-Strength Carbon-Steel Castings for General Application (A 281 – 45 T) (to be redesignated as Tent. Spec. A 27)	115	0	80
High-Strength Steel Castings for Structural Purposes (A 282 – 45 T) (to be redesignated as Tent. Spec. A 148)	105	0	90
III. TENTATIVE REVISION OF STANDARD			
Steel Music Spring Wire (A 228 – 41)	70	1	124
IV. ADOPTION OF TENTATIVE AS STANDARD			
Heat-Treated Carbon-and Alloy-Steel Track Bolts and Nuts (A 183 – 40 T), as revised	101	0	86
V. REVISION OF STANDARDS, IMMEDIATE ADOPTION			
Open-Hearth Carbon-Steel Rails (A 1 – 39)	80	0	107
Low-Carbon Steel Joint Bars (A 3 – 33)	81	0	106
Steel for Bridges and Buildings (A 7 – 42)	108	0	79
Structural Nickel Steel (A 8 – 39)	108	0	79
V. REVISION OF STANDARDS, IMMEDIATE ADOPTION—*Cont'd.*			
Wrought Steel Wheels for Electric Railway Service (A 25 – 41)	69	0	118
Boiler and Firebox Steel for Locomotives (A 30 – 44)	108	0	84
Welded and Seamless Steel Pipe (A 53 – 44)	116	0	71
Multiple-Wear Wrought Steel Wheels (A 57 – 44)	75	0	112
Steel Tie Plates (A 67 – 33)	82	0	105
Low-Carbon Steel Track Bolts and Nuts (A 76 – 33)	83	0	104
Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 – 44)	104	0	88
Structural Silicon Steel (A 94 – 39)	108	0	79
Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 105 – 40)	92	0	95
Commercial Quality Hot-Rolled Bar Steels (A 107 – 42)	113	2	80
Commercial Cold-Finished Bar Steels and Cold-Finished Shafting (A 108 – 36)	104	1	90
Structural Steel for Locomotives and Cars (A 113 – 42)	108	0	79
Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 – 44)	116	0	71
Open-Hearth Iron Plates of Flange Quality (A 129 – 39)	115	0	77
Structural Steel for Ships (A 131 – 39)	108	0	79
Electric-Resistance-Welded Steel Pipes (A 135 – 44)	116	0	71
Electric-Fusion-Welded Steel Pipe (Sizes 8 in. to but not Including 30 in.) (A 139 – 42)	116	0	71
Seamless Low-Carbon and Carbon-Molybdenum Steel Still Tubes for Refinery Service (A 161 – 44)	93	0	99
Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 44)	111	0	81
Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 – 44)	107	0	85
Forged or Rolled Steel Pipe Flanges for General Service (A 181 – 42)	93	0	94
Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 182 – 44)	92	0	95
One-Wear and Two-Wear Wrought Steel Wheels (A 186 – 39)	76	0	111
Carbon- and Alloy-Steel Nuts for Bolts for High-Pressure and High-Temperature Service (A 194 – 40)	91	1	103
Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 – 44)	105	0	87
Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 – 44)	105	0	87
Carbon-Silicon Steel Plates of Ordinary Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels (A 201 – 44)	115	0	77

TABLE III.—*Continued*

Items Specifications for the materials indicated	Affirmative	Negative	Ballots Marked "Not Voting"
V. REVISION OF STANDARDS, IMMEDIATE ADOPTION—*Cont'd.*			
Chrome-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels (A 202 - 44)	115	0	77
Low-Carbon Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 - 44)	101	0	86
Molybdenum-Steel Plates for Boilers and Other Pressure Vessels (A 204 - 44)	115	0	77
Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 209 - 44)	106	0	86
Medium-Carbon Seamless Steel Boiler and Superheater Tubes (A 210 - 44)	93	0	99
High Tensile Strength Carbon-Silicon Steel Plates for Boilers and Other Pressure Vessels (Plates 4½ in. and Under in Thickness) (A 212 - 44)	115	0	77
Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 - 44)	105	0	87
Electric - Resistance - Welded Steel Heat-Exchanger and Condenser Tubes (A 214 - 44)	98	0	89
Manganese-Vanadium Steel Plates for Boilers and Other Pressure Vessels (A 225 - 44)	115	0	77
Carbon-Steel Forgings for General Industrial Use (A 235 - 42)	101	0	94
Carbon-Steel Forgings for Locomotives and Cars (A 236 - 42)	101	0	94
Alloy-Steel Forgings for Locomotives and Cars (A 238 - 42)	102	0	90
Low-Alloy Structural Steel (A 242 - 42)	120	1	74
Atomic-Hydrogen-Arc-Welded and Electric-Resistance-Welded Alloy-Steel Boiler and Superheater Tubes (A 249 - 44)	94	0	93
Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 - 44)	93	0	99
Welded and Seamless Steel Pipe Piles (A 252 - 44)	83	0	104
Welded Alloyed Open-Hearth Iron Pipe (A 253 - 44)	116	0	71
VI. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS			
Steel for Bridges and Buildings (A 7 - 42)	120	0	75
Electric - Fusion - Welded Steel Pipe (Sizes 4 in. to but not Including 30 in.) (A 139 - 42)	103	0	89
Carbon- and Alloy-Steel Nuts for Bolts for High-Pressure and High-Temperature Service (A 194 - 40)	91	1	103
Carbon-Steel Forgings for General Industrial Use (A 235 - 42)	109	0	83
Carbon-Steel Forgings for Locomotives and Cars (A 236 - 42)	109	0	83
Alloy-Steel Forgings for General Industrial Use (A 237 - 42)	109	0	83
Alloy-Steel Forgings for Locomotives and Cars (A 238 - 42)	102	0	90
Low-Alloy Structural Steel (A 242 - 42)	120	0	75
Carbon-Steel and Alloy-Steel Ring and Disk Forgings (A 243 - 43)	109	0	83
Copper Brazed Steel Tubing (A 254 - 44)	90	0	102
VII. WITHDRAWAL OF STANDARDS			
Mild Steel Plates (A 10 - 39)	108	0	79
Carbon-Steel Castings for Miscellaneous Industrial Uses (A 27 - 44)	115	0	80
Carbon-Steel Plates for Stationary Boilers and Other Pressure Vessels (A 70 - 44)	112	1	74
Low Tensile Strength Carbon-Steel Plates of Structural Quality for Welding (A 78 - 43)	108	0	79
Carbon-Steel and Alloy-Steel Castings for Railroads (A 87 - 44)	115	0	80
Low Tensile Strength Carbon-Steel Plates of Flange and Firebox Qualities (A 89 - 43)	112	1	74
Alloy-Steel Castings for Structural Purposes (A 148 - 44)	105	0	90
Carbon-Steel Castings Suitable for Fusion Welding for Miscellaneous Industrial Uses (A 215 - 44)	115	0	80
Heat-Treated Wrought Steel Wheels (A 244 - 44)	75	0	112
VIII. ADOPTION AS STANDARD			
Carbon-Steel Plates 2 in. and Under in Thickness of Flange and Firebox Qualities, Low and Intermediate Tensile Strength (A 285 - 46) (To replace Specs. A 70 and A 89)	112	1	74
IX. REAFFIRMATION OF STANDARDS			
Open-Hearth Steel Girder Rails of Plain, Grooved and Guard Types (A 2 - 27)	81	0	106
Medium-Carbon Steel Joint Bars (A 4 - 14)	81	0	106
High-Carbon Steel Joint Bars (A 5 - 37)	81	0	106
Billet-Steel Bars for Concrete Reinforcement (A 15 - 39)	98	0	89
Rail-Steel Bars for Concrete Reinforcement (A 16 - 35)	98	0	89
Quenched Carbon-Steel Joint Bars (A 49 - 39)	81	0	106
Soft Steel Track Spikes (A 65 - 33)	81	0	106
Steel Screw Spikes (A 66 - 33)	81	0	106
Cold-Drawn Steel Wire for Concrete Reinforcement (A 82 - 34)	98	0	89
Cold-Rolled Strip Steel (A 109 - 38)	106	1	80
Axle-Steel Bars for Concrete Reinforcement (A 160 - 39)	98	0	89
Fabricated Steel Bar or Rod Mats for Concrete Reinforcement (A 184 - 37)	98	0	89
Welded Steel Wire Fabric for Concrete Reinforcement (A 185 - 37)	98	0	89
Hot-Worked High-Carbon Steel Tie Plates (A 241 - 41)	81	0	106

other possible steels which might be used, including some with higher chromium content, 1 per cent or over.

The revised Specifications for Alloy-Steel Bolting Materials for High-Temperature Service (A 193 - 45 T) now embody three grades formerly covered in the widely used Standard Specifications A 96 which do not give detailed chemical requirements, and with the approval of the revised tentative, the former Standard A 96 was withdrawn. (See note later in this report on additional changes in the bolting specifications.)

RECOMMENDATIONS AFFECTING STANDARDS

A large number of specifications affected by recommendations submitted in this report are listed in Table III, which also records the committee vote. Recommendations presented are detailed in the Appendix, and, where explanations of the proposals seem desirable, they are described in the sections of this report devoted to subcommittee activities.

NOTES ON SOME ACTIVITIES OF THE SUBCOMMITTEES, WITH REFERENCE TO SPECIFIC COMMITTEE RECOMMENDATIONS

Rails and Track Accessories:

Work involving specifications for steel rails and for track accessories, joint bars, track bolts, etc., is carried out in close cooperation with the American Railway Engineering Association, and an effort is made in so far as possible to have the actual requirements in the specifications of the two bodies coordinated. While this is not always completely possible, in general, the major requirements do coincide. This spirit of cooperation is one of the reasons for the numerous changes detailed in the Standard Specifications for Open-Hearth Carbon-Steel Rails (A 1 – 39) and in the Tentative Specifications for Heat-Treated Carbon- and Alloy-Steel Track Bolts and Nuts (A 183 – 40 T) and the Standard Specifications for Steel Tie Plates (A 67 – 33). The changes at the same time bring the requirements in line with the latest practice. Concurrent with this action, the wartime emergency provisions were withdrawn.

Structural Steel for Bridges, Buildings and Rolling Stock:

The most pressing problem confronting Subcommittee II has been the development of standard requirements for structural steel for welding. To coordinate the desires of the welding engineer for an absolute minimum of carbon and manganese, with physical properties in line with existing structural steel requirements for riveted construction; and the necessity from the mill practice angle of suitable chemical requirements in order to meet physical properties, has been a perplexing and much-discussed subject. Also there has not been agreement on the mechanics to be followed; that is, whether to embody the requirements in the widely-used Standard Specifications for Steel for Bridges and Buildings (A 7 – 42) or to promulgate separate specifications for structural steel for welding. After considerable debate and much correspondence in the special section headed by Jonathan Jones, it was agreed that separate specifications would be formulated which eventually might replace or become Standard Specification A 7.

In four of the structural specifications, A 7, A 8, A 94, and A 113, changes have been made in the scope clauses to clarify the use of the material. In A 7 a reference is being incorporated to the structural sheet specification A 245 so that the requirements of A 7 will not be used to cover thin gage material. As an editorial change, the section in A 7 referring to steel castings is being modified so that it will give up-to-date reference to the modified 65–35 grade in the revised castings specification A 27 – 46 T.

As a result of further deliberations at its meeting in Buffalo, N. Y. in June, 1946, the Section on Structural Steel for

Welding presented the following report which was approved by the subcomittee and Committee A-1:

1. That for the statically loaded parts of buildings A 7 steel is satisfactory up to 1½ in. thickness without special technique, and above that thickness can be kept so by proper technique.
2. That the same is true for most parts of highway bridges, assuming that a fair proportion of dead load stress is included in the total stress.
3. That for railway bridges A-7 steel is satisfactory up to 1 in. thickness without special technique, and above that thickness is likely to be of a composition necessitating pre-heat and possibly other special technique.
4. That there is a place for a specification for a steel for welded structures, the use of which should be considered when the thickness of a welded part is greater than 1½ in. in the case of buildings and highway bridges, and 1 in. in the case of railway bridges.
5. That one characteristic of this new specification would be the placing of a limit on carbon and manganese, permitting the strength to taper off with increase of thickness.
6. That it remains for bridge engineers to determine, and that the Section cannot determine whether any properties are essential in the steel which the present types of acceptance test do not measure.
7. That therefore the writing of this new specification will encounter some delay.
8. That when such a new specification is adopted, the Society should clearly distinguish, for the benefit of the consumer, where A-7 may be used and where the new specification is advised. This may well be done in the respective scope clauses, although it is not recommended that the scope clause of Specification A 7 be thus modified at this time.

To correlate the various thoughts in this field, there was formed last year a joint committee consisting of representatives of the American Welding Society, American Iron and Steel Institute, and the A.S.T.M., under the temporary chairmanship of F. H. Frankland. This joint committee is cooperating closely with Subcommittee II.

Two new specifications appended to this report[3]—one for Carbon-Steel Plates of Structural Quality With Low and Intermediate Tensile Strengths, 2 in. and under in Thickness; the other for Carbon-Silicon Steel Plates With Intermediate Tensile Strengths for Machine Parts and General Construction, over 2 in. in Thickness—will achieve certain objectives the subcommittee has had in mind for some time. The first named specification essentially is a consolidation of two existing standards covering Mild Steel Plates (A 10 – 39) and Low Tensile Strength Carbon-Steel Plates of Structural Quality for Welding (A 78 – 43). The new specification covers in one document the three commonly used grades and this channeling of production should be beneficial to both the consumer and producer. In combining Specifications A 78 and A 10, some features which differed in the two standards have been reconciled. The thickness is limited to 2 in. since it is felt the separate specification with silicon content is more appropriate, and forge welding quality steel has been dropped since this process is almost obsolete. However, in specifications that are being recommended by Subcommittee XI on Steel for Pressure Vessels this class is continued.

The new specification for Carbon-Silicon Steel Plate meets the need for grades of steel to be rolled in heavier thicknesses than 2 in. but the requirements for which are not as rigorous as in the Specifications for Carbon-Silicon Steel Used for Boilers and Other Pressure Vessels, A 201 and A 212 respectively.

These new specifications incorporate statements and requirements with respect to surface finish and conditioning and permissible variations in weight and thickness which are believed to bring the specifications in line with latest thoughts and industrial practices.

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[3] These specifications were accepted as tentative by the Society and appear in the 1946 Book of A.S.T.M. Standards, Part I-A.

Music Spring Wire:

The revised table of tensile requirements shown in the Appendix, involving the Standard Specifications for Music Spring Wire (A 228 – 41) resulted from intensive studies that had been undertaken to eliminate an abnormality in current values, particularly in the smaller sizes of wire. When the values were plotted, instead of a reasonably straight-line curve which might be expected, there was a very noticeable departure below about 20 to 30 mils. A number of revised values were proposed by different committee members, and two sets of these seemed to represent a good average. These were studied mathematically with methods drawn from probability theory, and using an assumed form of equation a formula was developed which could be used in calculating the proposed values shown in Table IV in the Appendix.

Concerning the size of wire to be covered, the present maximum of 0.156 in. has been retained, since it seemed to a number of those concerned that anything larger would be outside of what is considered commercially as music wire.

Subcommittee IV is studying the possibility of establishing two different qualities of hard-drawn wire now covered in the Standard Specifications for Hard-Drawn Steel Spring Wire (A 227 – 41) and also is investigating the demand for new specifications intermediate between hard-drawn and music spring wire.

Concrete Reinforcement Steel:

Although there was agreement to withdraw the Emergency Alternate EA – A 160, Specification for Axle-Steel Bars for Concrete Reinforcement, which provided during wartime for use of a wider range of materials than covered in the normal specification for axle-steel bars, the committee acted to start drafting a new specification for bars rolled from material where the heats are not identified.

Carbon- and Alloy-Steel Forgings:

With the transfer of the several emergency specifications for heavy forgings (ES-21 to ES-27 incl.) to the status of tentative, the withdrawal of a number of emergency alternate provisions in the other forgings specifications, plus some minor revisions, and the adoption as standard of tentative revisions which were issued in August, 1944, the specifications under the jurisdiction of Subcommittee VI are considered up to date. Certain studies are under way in the committee involving macroetch testing and the incorporation of such requirements in specifications. There is evident a need of standardization and simplification, but how to set up a series of pictorial representations of good practice or which might be considered "go or no go" practice, entails much study. Such pictorial standards have been evolved for application in a rather narrow field by Watertown Arsenal. Some progress is being made, but the final answer will necessarily be slow in forthcoming.

A Special Section on Hardness Requirements has developed some general principles which should be considered in incorporating requirements on hardness in forgings specifications. These principles follow:

(*A*) Hardness tests, when required, should be specified under the following conditions:

(*1*) Brinell hardness may be required only when tensile properties are not specified.

(*2*) For special forgings, such as, pinion gears, reduction gears, and similar forgings where surface hardness is a factor and may differ from the hardness indicated by the tensile properties, both Brinell hardness number and tensile properties may be specified upon agreement between the purchaser and producer.

(*B*) The following principles should apply in making the hardness test:

(*1*) The accepted method for hardness testing is the standard Brinell method, and it should be used in cases where size of forging permits.

(2) Where size of forging will not permit the use of the standard Brinell method, the use of any other methods should be governed by the comparable permissible working range specified for the Brinell method.

(3) A hardness range should be specified only for quenched-and-tempered or normalized-and-tempered forgings. For normalized or annealed forgings, a maximum or a minimum figure only should be specified. No hardness specification should be applied to untreated forgings.

Rolled Steel Wheels and Steel Tires:

The revised Specification for Multiple-Wear Wrought-Steel Wheels[4] is a revision and combination of two existing specifications: one covering heat-treated wheels of three classes (A 244), and the other, a nonheat-treated grade (A 57). There has been sufficient use of the standards so that the combination is considered feasible. The new specification will simplify ordering and production, and will bring the requirements substantially in line with those of the American Association of Railroads. Committee A-1 cooperates closely with the A.A.R. in various phases of the specifications work on steel wheels.

Revisions in the Standard Specifications for One-Wear and Two-Wear Wrought-Steel Wheels (A 186 – 39) involving primarily the cancellation of the two-wear type are justified because of lack of production of the latter.

Experience with the Emergency Alternate Provisions (EA – A 25a) in the Standard Specifications for Wrought-Steel Wheels for Electric Railway Service (A 25 – 41) justify the retention of some of the requirements as regular provisions.

Rolled Carbon-Steel Locomotive Frames:

Since the material covered in the new Tentative Specifications for Rolled Carbon-Steel Locomotive Frames[3] did not logically fall in the province of any of the standing subcommittees, a special group of users and producers was established. After full consideration of the problem involved, agreement was reached on requirements and tests and the proposed new tentative specifications embody the results.

Steel Castings:

In November, 1945, there were issued two new Tentative Specifications for Mild- to Medium-Strength Carbon-Steel Castings for General Application (A 281 – 45 T) and High-Strength Steel Castings for Structural Purposes (A 282 – 45 T). The first named is essentially a consolidation of three existing specifications, A 27, A 87 and A 215, while the latter brings up to date the requirements in the Standard Specifications A 148 of which further details are given earlier in this report. The committee in charge in proposing additional changes in the specifications, as noted in the Appendix, is very anxious to have the older designations which are so widely known in the industry, apply to these new tentatives and therefore, instead of A 281 the designation of A 27 is to apply, and for A 282 the designation A 148 is to be reassigned. This contemplates the withdrawal of the four standards essentially combined in the new tentatives.

Steel Pipe and Tubing:

(See also Notes on Materials for High-Temperature Service)

While probably none of the recommendations in the large number of pipe and tubing specifications under the jurisdiction of Subcommittee IX are of major concern, they all are in line with this very active subcommittee's constant endeavor to have the specifications up to date and in line with current practices, and to meet, in so far as possible, the needs of the consumer. Some of the material being incorporated in the specifications, particularly in respect to chemical composition, comes from the desire to set up as peacetime practice, limitations which were in effect during the war period as emergency alternate provisions.

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[4] This revised specification was accepted by the Society and appears in the 1946 Book of A.S.T.M. Standards, Part I-A.

One change which affects a large number of the pipe and tubing specifications where thin walls are produced merits discussion. In these several specifications a set of formulas was incorporated a few years ago to give the required values of elongation for strip tests cut from material less than $\frac{5}{16}$ in. in thickness. The use of these values has brought to light numerous cases where the existing rule has caused the rejection of light wall material which failed to meet the elongation requirements for the next heavier wall. Consequently a formula has been developed to cover wall thicknesses lying between the values now shown in the specifications. This subcommittee has several studies under way one of which involves requirements for pipe and tubing for low-temperature service.

Since a conference of those concerned with heat exchange and still tubes indicated the time would be propitious for eliminating the dual system of designating wall thicknesses in several A-1 specifications, and to standardize on the minimum wall requirements, favorable action was taken to delete from six still tube and heat exchanger specifications all references to average wall, and to have the requirements refer only to minimum wall thicknesses. Specifications affected include A 161, A 200, A 271, A 179, A 199, and A 214.

To clarify requirements in various pipe specifications, and to emphasize that these requirements are based on nominal (average) wall thickness, numerous editorial changes are being incorporated in the specifications listed below. To be added to each of these specifications is a detailed table of minimum wall thicknesses for the corresponding nominal (average) thickness. So that there should be a minimum of misunderstanding about this new table, a note is to accompany it indicating that the use of the table is no assurance that all of the thicknesses or sizes listed can be obtained.

A 53
A 106
A 120
A 135
A 139
A 158
A 206
A 252
A 253
A 280

Steel for Boilers and Pressure Vessels:

The two specifications covering Low Carbon-Nickel-Steel Plates for Boilers and Other Pressure Vessels,[5] and Carbon-Steel Plates 2 in. and under in Thickness of Flange and Firebox Qualities, Low and Intermediate Tensile Strength,[5] are recommended to bring up to date existing requirements for these materials. Nickel steel plates have been ordered in thicknesses up to 6 in., and the existing Specification A 203 is limited in application to 2 in. The second new specification is intended as a consolidation of two existing standards, Specifications for Carbon-Steel Plates for Stationary Boilers and Other Pressure Vessels (A 70 – 44), which material is suitable for fusion welding; and Specifications for Low Tensile Strength Carbon-Steel Plates of Flange and Firebox Qualities (A 89 – 43). It is believed that the combined specifications will be advantageous, giving in one document coverage of material that can be used for different types of vessels, for riveting, for fusion welding, and firebox quality for forge welding. A number of modifications in the requirements provided in the proposed specifications have been considered by the subcommittee in charge. Decision will be reached on whether the existing standards are to be withdrawn with the approval of the new items or whether they will be carried for one year.

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[5] These revised specifications were accepted by the Society and appear in the 1946 Book of A.S.T.M. Standards, Part I-A.

In several standards there is to be a clarification of whether the rolling mill or the fabricator will perform the required heat treatment.

Revisions proposed for immediate adoption in the Standard Specifications for Boiler and Firebox Steel for Locomotives (A 30 – 44) will bring the firebox grades in line with recent changes in A.A.R. specifications.

Affecting most of the standards in the charge of this subcommittee is a proposed revision which will permit the buyer to specify on his order that test specimens are to be stress-relieved at a specified temperature, and the physical results reported in this condition.

Methods of Physical Test

Based on studies and recommendations carried out in Subcommittee XIII on Methods of Physical Tests, revisions have been approved in the Methods of Tension Testing of Metallic Materials, E 8, which clarify the requirements and should tend to eliminate misunderstandings about size, design of certain specimens, and related matters.

Consideration was given to the reorganization of Subcommittee XIII and a statement of policy on which it should base any actions with the following recommendations was approved:

> It is recommended, as a matter of policy, that Subcommittee XIII act for Committee A-1 in establishing Committee A-1 methods of physical and mechanical testing. It is further recommended that the origin and responsibility of such methods should rest with the individual A-1 subcommittees who have specific products in their charge. (Subcommittee XIII thus acting in a clearing and advisory capacity.)
>
> The several product committees of A-1 are requested to proceed immediately with the review and preparation of test procedures of their own and submit their ideas to Subcommittee XIII for study.

Recognizing that as the work of Subcommittee XIII develops, it might be advisable to organize Subcommittee XIII into several sections (with possibly an addition to its membership), the Subcommittee authorized the chairman to confer with A.S.T.M. Headquarters in the development of the program respecting the organization of Subcommittee XIII.

Carbon- and Alloy-Steel Bars:

There has been a steadily growing conviction that Committee A-1 would render considerable service to many users of bar steels if requirements could be agreed on to provide specified physical properties to cover the bars in various thermal conditions. While great tonnages of bar material may not be the rule in such industries as electrical manufacturing, railroad, petroleum refining and many others, as is the case for example in the automotive field, nevertheless there is a very significant production and the design engineers and others in different consuming fields have felt the need for material with specified physical properties. Consequently the new Tentative Specifications for Heat-Treated Alloy-Steel Bars[3] is a step in the general direction toward which the Subcommittee XV on Bar Steels is moving. It is hoped that in the not too far distant future other specifications may be forthcoming, including one on hot-rolled bars subject to tensile requirements, another on hot-rolled annealed and heat-treated carbon bars, and possibly still another for alloy-steel bars with chemical requirements which might embody hardenability provisions.

The revisions in the existing commercial bar specifications, hot-rolled (Specifications A 107)[5] and cold-finished (Specifications A 108)[5] will bring these requirements up to date. They also incorporate as information tables of estimated tensile values and suggested applications.

Sheet Steel and Steel Sheets:

There are three specifications in this field: one covering cold-rolled strip (Specifications A 109) and two others covering light-gage, flat rolled steel for structural applications (Specifications A 245 and A 246). Changes are pending in the chemical requirements and check analysis for strip, and also in the other two specifications where the bending properties are a subject of study. Minor changes clarifying the use of the bend test specimen have been recommended in each specification.

A new project confronting this committee is the development of requirements on hot-rolled strip steel which can be used in structural applications. Previously it has been possible to use some of the requirements in the structural steel specification A 7 but the revision now limits the use of A 7 to thicker material. Subcommittee XIX is drafting a hot-rolled strip specification.

Valves, Fittings, Piping and Flanges for High-Temperature Service:

Reference has been made earlier in this report to the problem of graphitization of carbon-molybdenum pipe at temperatures above 900 to 925 F. New Tentative Specifications covering chromium-molybdenum pipe have been issued. This subcommittee is studying the effect of possible graphitization upon other materials and piping, including certain castings and forged fittings.

The Tentative Specifications for Alloy-Steel Bolting Materials for High-Temperature Service (A 193 - 45 T) have been studied, particularly for chemical composition of the various grades included, and the new requirements include permissible variations on check analysis. This entails some more rigid restrictions on the ladle analysis limits.

To clarify the intent that flanges must not be cut or machined from plate as made in accordance with the three following specifications covering flange material for normal and for elevated temperature service, the section on manufacturing practice has been modified. Specifications affected are A 181, A 182, and A 105.

Subcommittee XXII and also Subcommittee IX on Pipe and Tubing were concerned with the use of deoxidized bessemer steel for service at elevated temperatures, particularly where some forming operations were involved. Several investigations were undertaken to evaluate this material, which is produced in large tonnages, and so the results of the investigations will be available for future reference, it is hoped the material can be published in an ensuing report of Committee A-1.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, N. L. Mochel.
Vice-Chairman, H. B. Oatley.
Vice-Chairman, T. G. Stitt.
Secretary, Henry Wysor.*

This report has been submitted to letter ballot of the committee, which consists of 258 members; 215 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

N. L. MOCHEL,
Chairman.

R. J. PAINTER,
Secretary.

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*Appointed by Committee A-1 in January, 1947.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee A-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Revision of Standard Specifications for:

Electric-Resistance-Welded and Open-Hearth Iron Boiler Tubes (A 178 - 44),
Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 - 44),
Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 - 44),
Atomic-Hydrogen-Arc-Welded and Electric-Resistance-Welded Alloy-Steel Boiler and Superheater Tubes (A 249 - 44), and
Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 - 44).

These recommendations were accepted by the Standards Committee on July 22, 1946, and the tentative revisions appear in the 1946 Book of A.S.T.M. Standards, Part I-A.

The emergency provisions listed in Table I of the Report were discontinued by action of the Standards Committee on September 9, 1946.

On January 14, 1947, the Administrative Committee on Standards accepted the recommendation of Committee A-1 that the following specifications and method of test be revised:

Tentative Specifications for:

Carbon-Chromium Ball and Roller-Bearing Steels (A 295 - 46 T).

Tentative Method of:

End-Quench Test for Hardenability of Steel (A 255 - 46 T).

These revised tentatives appear in the 1946 Book of A.S.T.M. Standards, Part I-A bearing the designations indicated above.

On February 3, 1947 the Administrative Committee on Standards approved the revisions submitted by Committee A-1 in the Tentative Specifications for Light Gage Structural Quality Flat Hot-Rolled Carbon Steel (0.2499 and 0.1874 in. to 0.0478 in. in Thickness) (A 245 - 47 T), and the Tentative Specifications for Light Gage Structural Quality Flat Rolled Carbon Steel (0.0477 to 0.0225 in. in Thickness) (A 246 - 47 T). These revised tentative specifications have been issued separately in reprint form.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS ON STEEL

In this Appendix recommendations are given affecting certain specifications, both standard and tentative, covering various steel products. These specifications appear in their present form in either the 1944 Book of A.S.T.M. Standards, Part I, or the 1945 Supplement, Part I, as indicated by the final number in the A.S.T.M. designation.

TABLE I.—PERMISSIBLE OVERWEIGHTS OF PLATES ORDERED TO THICKNESS.

Specified Thickness, in.	Permissible Excess in Average Weight of Lots[a] for Widths Given in Inches, Expressed in Percentage of Nominal Weights										
	48 and under	Over 48 to 60, excl.	60 to 72, excl.	72 to 84, excl.	84 to 96, excl.	96 to 108, excl.	108 to 120, excl.	120 to 132, excl.	132 to 144, excl.	144 to 168, excl.	168 and over
3/16 to 1/4, excl.	...	8	9	10	12	14	16	18	...	...	...
1/4 to 5/16, excl.	6	7	8	9	10	12	14	16	19	...	...
5/16 to 3/8, excl.	5	6	7	8	9	10	12	14	17	18	...
3/8 to 7/16, excl.	4.5	5	6	7	8	9	10	12	15	16	18
7/16 to 1/2, excl.	4	4.5	5	6	7	8	9	10	13	14	16
1/2 to 5/8, excl.	4	4	4.5	5	6	7	8	9	11	12	14
5/8 to 3/4, excl.	4	4	4	4.5	5	6	7	8	9	10	12
3/4 to 1, excl.	3.5	4	4	4	4.5	5	6	7	8	9	11
1 to 2, incl.	3.5	3.5	4	4	4	4.5	5	6	7	8	9

[a] The term "lot" means all the plates of each tabular width and thickness group represented in each shipment.

NOTE 1.—Permissible variation under specified thickness, 0.01 in.

NOTE 2.—Permissible variations in weight for single plates shall be 1⅓ times the amounts given in the table.

NOTE 3.—Permissible overweight for circular and sketch plates shall be 25 per cent greater than the amounts given in the table.

NOTE 4.—The adopted standard density for rolled steel is 0.2833 lb. per cu. in.

TABLE II.—PERMISSIBLE VARIATIONS OF PLATES ORDERED TO WEIGHT.

Specified Weights, lb. per sq. ft.	Permissible Variation in Average Weight of Lots[a] for Widths Given in Inches, Expressed in Percentages of the Specified Weights (Pounds per Square Foot)																					
	48 and under		Over 48 to 60 excl.		60 to 72, excl.		72 to 84, excl.		84 to 96, excl.		96 to 108, excl.		108 to 120, excl.		120 to 132, excl.		132 to 144, excl.		144 to 168, excl.		168 and over	
	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under	Over	Under
7.65 to 10, excl.	...	...	4.5	3	5	3	5.5	3	6	3	...	...	...	...	...	...	...	...	...	...	...	...
10 to 12.5, excl.	4	3	4.5	3	5	3	5.5	3	6	3	6.5	3	7	3	8	3	9	3	...	...	...	...
12.5 to 15, excl.	4	3	4	3	4.5	3	5	3	5.5	3	5.5	3	6	3	7.5	3	8	3	...	...	...	...
15 to 17.5, excl.	3.5	3	3.5	3	4	3	4.5	3	5	3	5	3	5.5	3	6	3	7	3	9	3	10	3
17.5 to 20, excl.	3.5	2.5	3.5	2.5	3.5	3	4	3	4.5	3	4.5	3	5	3	5.5	3	6	3	8	3	9	3
20 to 25, excl.	3.5	2.5	3.5	2.5	3.5	3	3.5	3	4	3	4	3	4.5	3	5	3	5.5	3	7	3	8	3
25 to 30, excl.	3	2.5	3.5	2.5	3.5	2.5	3.5	3	3.5	3	3.5	2.5	4	3	4.5	3	5	3	6.5	3	7	3
30 to 40, excl.	3	2	3	2	3	2	3	2	3.5	2	3.5	2	3.5	2.5	4	3	4.5	3	6	3	6.5	3
40 to 81.6, incl.	2.5	2	3	2	3	2	3	2	3.5	2	3.5	2	3	2.5	3.5	3	4	3	5.5	3	6	3

[a] The term "lot" means all the plates of each tabular width and weight group represented in each shipment.

NOTE 1.—Permissible variations in weight for single plates shall be 1⅓ times the amounts given in the table.

NOTE 2.—Permissible overweight for circular and sketch plates shall be 25 per cent greater than the amounts given in the table.

NOTE 3.—The adopted standard density for rolled steel is 0.2833 lb. per cu. in.

Immediate Adoption of Requirement on Stress Relieving Test Specimens.—In the following seven specifications for plates for boilers and pressure vessels insert a paragraph in the respective sections covering test specimens, making the necessary editorial changes in the other paragraphs:

A 30 - 44
A 129 - 39
A 201 - 44
A 202 - 44
A 204 - 44
A 212 - 44
A 225 - 44

This paragraph is to read as follows:

When so specified on the purchase order with the mill, test specimens representing the plates shall be stress relieved at the mill at a temperature of 1150 F. (or at a temperature otherwise agreed upon), tested, and the physical results reported in this condition.

Immediate Adoption of Revised Permissible Variations in Thickness and Weight.—In the following 13 specifications revise the tables of permissible overweights of plates ordered to thickness, variations of plates ordered to weight, and variations over ordered thickness of plates over 2 in. in thickness to be in accordance with the accompanying Tables I, II, and III.

NOTE.—Depending on the scope of the particular specifications some of the tabular material will not apply and accordingly will be editorially revised.

A 7 - 42
A 8 - 39
A 30 - 44
A 94 - 39
A 113 - 42
A 129 - 39
A 131 - 39
A 201 - 44
A 202 - 44
A 204 - 44
A 212 - 44
A 225 - 44
A 242 - 42

Immediate Adoption of Revised Bend Test Procedure.—In the following six specifications revise the section on bend test procedure and the bend test requirement table to eliminate the use of the pin.

A 7 - 42
A 8 - 39
A 94 - 39
A 113 - 42
A 131 - 39
A 242 - 42

REVISIONS OF TENTATIVES

Tentative Specifications for Seamless Carbon-Steel Pipe for High-Temperature Service (A 106 - 45 T):

Section 1(a).—Change the second sentence to read as follows by the addition of the italicized words: "Pipe ordered under these specifications is *nominal (average) wall and* shall be suitable for bending, flanging, and similar forming operations (see Explanatory Note 2)."

Section 2(a).—Delete the phrase "silicon killed" after the reference to grade B in the second line.

TABLE III.—PERMISSIBLE VARIATIONS OVER ORDERED THICKNESS OF PLATES OVER 2 IN. IN THICKNESS.

Specified Thickness, in.	Variations over Specified Thickness For Widths Given, in.					
	Up to 36, excl.	36 to 60, excl.	60 to 84, excl.	84 to 120, excl.	120 to 132, excl.	132 and over
Over 2 to 3, excl.	1/16	3/32	7/64	1/8	1/8	9/64
3 to 4, excl.	5/64	3/32	7/64	1/8	1/8	9/64
4 to 6, excl.	3/32	1/8	9/64	9/64	5/32	11/64
6 to 8, excl.	7/64	1/8	5/32	5/32	11/64	...
8 to 10 excl.	...	11/64	3/16	3/16	...	...
10 to 12 excl.	...	3/16	15/64	15/64	...	...

NOTE 1.—Permissible variation under specified thickness, 0.01 in.

Table I.—Delete the phrase "silicon killed" after "Grade B" in the heading for column 3. Change the maximum carbon content requirement for grade B from the present "0.35" to "0.30" per cent and add a minimum silicon content requirement of "0.10" for grade A.

Section 7.—Change the value in the flattening test formula for the constant *e* for grade A from the present "0.09" to "0.08."

Table II.—At the end of this table add the following note:

NOTE.—The above table gives the computed minimum elongation values for each $\frac{1}{32}$ in. decrease in wall thickness. Where the wall thickness lies between two values shown above, the minimum elongation value shall be determined by the following formula:

Grade	Direction of Test	Formula
A	Longitudinal.......	$E = 56t + 17.50$
A	Transverse.........	$E = 40t + 12.50$
B	Longitudinal.......	$E = 48t + 15.00$
B	Transverse.........	$E = 32t + 6.50$

where:

E = elongation in 2 in. in per cent, and
t = actual thickness of specimen, in inches.

Section 13.—Add a note at the end of this section to read as follows: "The minimum wall thickness on inspection is shown in Table V (Appendix II)."

Table IV (Appendix I).—Enter recalculated values in column 5 for grade A due to the change in the constant e in the flattening test formula.

New Table:—Add a new Table V as Appendix II to read as shown in the accompanying Table IV.

TABLE IV.—TABLE OF MINIMUM WALL THICKNESSES ON INSPECTION FOR NOMINAL (AVERAGE) PIPE WALL THICKNESSES.

NOTE 1.—The following formula, upon which this table is based, may be applied to calculate minimum wall thickness from nominal (average) wall thickness:

$$t_n \times 0.875 = t_m$$

where:

t_n = nominal (average) wall thickness in inches, and
t_m = minimum wall thickness in inches.

The wall thickness is expressed to three decimal places, the fourth decimal place being carried forward or dropped, in accordance with the Tentative Recommended Practices for Designation of Numerical Requirements in Standards (A. S.T.M. Designation: E 29).

NOTE 2.—This table is a master table covering wall thicknesses available in the purchase of different classifications of pipe, but it is not meant to imply that all of the walls listed therein are obtainable under this specification.

Nominal (Average) Thickness (t_n), in.	Minimum Thickness on Inspection (t_m), in.	Nominal (Average) Thickness (t_n), in.	Minimum Thickness on Inspection (t_m), in.	Nominal (Average) Thickness (t_n), in.	Minimum Thickness on Inspection (t_m), in.
0.068	0.060	0.279	0.244	0.600	0.525
0.088	0.077	0.280	0.245	0.625	0.547
0.091	0.080	0.281	0.246	0.656	0.574
0.095	0.083	0.294	0.257	0.674	0.590
0.113	0.099	0.300	0.262	0.687	0.601
0.119	0.104	0.307	0.269	0.718	0.628
0.125	0.109	0.308	0.270	0.750	0.656
0.126	0.110	0.312	0.273	0.812	0.710
0.133	0.116	0.318	0.278	0.843	0.738
0.140	0.122	0.322	0.282	0.864	0.756
0.145	0.127	0.330	0.289	0.875	0.766
0.147	0.129	0.337	0.295	0.906	0.793
0.154	0.135	0.343	0.300	0.937	0.820
0.156	0.136	0.344	0.301	1.000	0.875
0.179	0.157	0.358	0.313	1.031	0.902
0.187	0.164	0.365	0.319	1.062	0.929
0.188	0.164	0.375	0.328	1.125	0.984
0.191	0.167	0.382	0.334	1.156	1.012
0.200	0.175	0.400	0.350	1.218	1.066
0.203	0.178	0.406	0.355	1.250	1.094
0.216	0.189	0.432	0.378	1.312	1.148
0.218	0.191	0.436	0.382	1.343	1.175
0.219	0.192	0.437	0.382	1.406	1.230
0.226	0.198	0.438	0.383	1.437	1.257
0.237	0.207	0.500	0.438	1.500	1.312
0.250	0.219	0.531	0.465	1.562	1.367
0.258	0.226	0.552	0.483	1.750	1.531
0.276	0.242	0.562	0.492		
0.277	0.242	0.593	0.519		

Tentative Specifications for Seamless Alloy-Steel Pipe for High-Temperature Service (A 158 – 44 T):

Table I.—To bring the manganese ranges in line with manufacturers' practices and A.I.S.I. compositions, in grades P 5a, P 5b and P 5c change the required manganese content from the present "0.50 max." to read "0.30 to 0.60" per cent.

In grades P 3a and P 11 change the required maximum phosphorus content from the present "0.04" to read "0.03" per cent and change the required maximum sulfur content from the present

"0.05" to read "0.03" per cent; also change the required manganese content of grade P 3a from the present "0.40 to 0.60" to read "0.30 to 0.60" per cent.

In this same table, add two new grades, a 7 per cent chromium and a 9 per cent chromium, which had appeared on EA - 158a, with chemical requirements to read as follows:

	P 16 7 per cent Chromium	P 17 9 per cent Chromium
Carbon, max., per cent....	0.15	0.15
Manganese, per cent.....	0.30 to 0.60	0.30 to 0.60
Phosphorus, max., per cent	0.03	0.03
Sulfur, max., per cent.....	0.03	0.03
Silicon, per cent..........	0.50 to 1.00	0.50 to 1.00
Nickel, per cent..........	......	......
Chromium, per cent.......	6.00 to 8.00	8.00 to 10.00
Molybdenum, per cent....	0.45 to 0.65	0.90 to 1.10

Section 1(b).—Change the first sentence to read as follows by the addition of the italicized words: "These specifications cover *nominal (average) wall* seamless alloy-steel pipe intended for high-temperature service."

Table II.—Add the same note to Table II as recommended above for Table II in Specifications A 106, the formulas to read as follows:

Grade	Direction of Test	Formula
Ferritic	Longitudinal	$E = 48t + 15.00$
Ferritic	Transverse	$E = 32t + 10.00$
Austenitic	Longitudinal	$E = 80t + 25.00$
Austenitic	Longitudinal	$E = 64t + 20.00$

Section 16.—Add a note at the end of this section to read as follows: "Note.—The minimum wall thickness on inspection is shown in Table III (Appendix II)."

New Table.—Add a new Table III as Appendix II to read as shown in the accompanying Table IV.

Tentative Specifications for Alloy-Steel Bolting Materials for High-Temperature Service (A 193 – 45 T):

Section 1.—Add a new Paragraph (*c*) to read as follows, relettering the present Paragraph (*c*) as (*d*):

(*c*) To meet the requirements of grades BA, BB and BC, the following types of alloy steel may be submitted: nickel, chromium-nickel, chromium - vanadium, chromium - manganese, chromium - molybdenum, nickel - chromium - molybdenum, or any other types approved by the purchaser.

Table I.—Change the chemical composition requirements for grade B7a (chromium-high molybdenum) to read as prescribed in the following table:

	Present Values	Revised Values
Carbon, per cent..........	0.35 to 0.45	0.35 to 0.50
Manganese, per cent......	0.60 to 0.90	0.70 to 1.00
Phosphorus, max., per cent.	0.04	0.04
Sulfur, max., per cent......	0.05	0 05
Silicon, per cent...........	0.15 to 0.30	0.18 to 0.37
Nickel, per cent	......	......
Chromium, per cent	0.80 to 1.10	0.75 to 1.15
Molybdenum, per cent....	0.45 to 0.65	0.45 to 0.65
Tungsten, per cent	......	......
Vanadium, per cent.......	......	......

Also add requirements for check variations to Table I so that the revised table will read as shown in the accompanying Table V.

Table II.—Change the tensile requirements for grade B7a (chromium-high molybdenum) to read as follows:

Diameter, in.	Minimum Tempering Temperature, deg. Fahr.	Tensile Strength, min., psi.	Yield Point, min., psi.	Elongation in 2 in., min., per cent	Reduction of Area, min., per cent
2½ and under.....	1000	140 000	120 000	15	45
	1100	135 000	115 000	15	50
	1200	**125 000**	**105 000**	**16**	**50**
Over 2½ to 4, incl.	1000	130 000	110 000	15	45
	1100	125 000	105 000	15	50
	1200	115 000	95 000	16	55

Revise the requirements for grades BA, BB and BC in Table II to read as shown in the accompaning Table V-A.

Tentative Specifications for Seamless Carbon-Molybdenum Alloy-Steel Pipe for High-Temperature Service (A 206 – 44 T):

Section 1(a).—Change the first sen-

TABLE V.—CHEMICAL COMPOSITION.

Type	Ferritic Steels					
Identification Symbol	BA		BB		BC	
	Range	Check Variation, Over[g]	Range	Check Variation, Over[g]	Range	Check Variation, Over[g]
Phosphorus, max., per cent	0.04	0.005	0.04	0.005	0.04	0.005
Sulfur, max., per cent	0.04	0.005	0.04	0.005	0.04	0.005

Type	Ferritic Steels					
Identification Symbol	B4		B5		B6	
Grade	Nickel-Chromium-Molybdenum		5 per cent Chromium (A.I.S.I. Type 501 Modified)		12 per cent Chromium (A.I.S.I. Type 416)	
	Range	Check Variation, Over or Under[g]	Range	Check Variation, Over or Under[g]	Range	Check Variation, Over or Under[g]
Carbon, per cent	0.38 to 0.45	0.02	0.10 to 0.30	0.02	0.15 max.	0.01 over
Manganese, per cent	0.55 to 0.75	0.03	0.30 to 0.60	0.03	1.25 max.	0.04 over
Phosphorus, max., per cent	0.04	0.005 over	0.04	0.005 over	0.14[d, f]	[a]
Sulfur, max., per cent	0.04	0.005 over	0.03	0.005 over	0.50[b, d, f]	[a]
Silicon, per cent	0.20 to 0.35	0.02	0.30 to 0.60	0.05	0.30 to 0.60	0.05
Nickel, per cent	1.60 to 1.95	0.05	....	....	....	....
Chromium, per cent	0.55 to 0.75	0.03	4.00 to 6.00	0.10	12.00 to 14.00	0.15
Molybdenum, per cent	0.30 to 0.40	0.02	0.45 to 0.65	0.05	[e]	0.03 over
Tungsten, per cent	....	....	....	....	....	....
Vanadium, per cent	....	....	....	....	....	....
Selenium, per cent	....	....	....	....	0.50 max.[b, f]	0.03 over
Zirconium, per cent	....	....	....	....	[e]	....

Type	Ferritic Steels					
Identification Symbol	B7		B7a		B11	
Grade	Chromium-Molybdenum (A.I.S.I. 4140 or 4142)		Chromium-High Molybdenum		Tungsten-Chromium-Vanadium	
	Range	Check Variation, Over or Under[g]	Range	Check Variation, Over or Under[g]	Range	Check Variation, Over or Under[g]
Carbon, per cent	0.38 to 0.45	0.02	0.38 to 0.45	0.02	0.41 to 0.48	0.02
Manganese, per cent	0.75 to 1.00	0.04	0.75 to 1.00	0.04	0.25 to 0.40	0.03
Phosphorus, max., per cent	0.04	0.005 over	0.04	0.005 over	0.04	0.005 over
Sulfur, max., per cent	0.04	0.005 over	0.04	0.005 over	0.04	0.005 over
Silicon, per cent	0.20 to 0.35	0.02	0.20 to 0.35	0.02	0.29 to 0.35	0.02
Nickel, per cent	....	....	....	....	....	....
Chromium, per cent	0.80 to 1.10	0.05	0.80 to 1.10	0.05	1.00 to 1.50	0.05
Molybdenum, per cent	0.15 to 0.25	0.02	0.50 to 0.70	0.05	....	....
Tungsten, per cent	....	....	....	....	1.70 to 2.30	0.10
Vanadium, per cent	....	....	....	....	0.20 to 0.30	0.03
Selenium, per cent	....	....	....	....	....	....
Zirconium, per cent	....	....	....	....	....	....

Type	Ferritic Steels					
Identification Symbol	B12		B13		B14	
Grade	Nickel-Chromium (A.I.S.I. 3140)		Tungsten-Molybdenum-Chromium		Chromium-Molybdenum-Vanadium	
	Range	Check Variation, Over or Under[g]	Range	Check Variation, Over or Under[g]	Range	Check Variation, Over or Under[g]
Carbon, per cent	0.38 to 0.45	0.02	0.38 to 0.45	0.02	0.41 to 0.48	0.02
Manganese, per cent	0.70 to 0.90	0.03	0.75 to 1.00	0.04	0.45 to 0.65	0.03
Phosphorus, max., per cent	0.04	0.005 over	0.04	0.005 over	0.04	0.005 over
Sulfur, max., per cent	0.04	0.005 over	0.04	0.005 over	0.04	0.005 over
Silicon, per cent	0.20 to 0.35	0.02	0.20 to 0.35	0.02	0.20 to 0.35	0.02
Nickel, per cent	1.10 to 1.40	0.05	....	....	....	....
Chromium, per cent	0.55 to 0.75	0.03	0.50 to 0.70	0.03	0.80 to 1.10	0.05
Molybdenum, per cent	....	....	0.45 to 0.65	0.05	0.30 to 0.40	0.02
Tungsten, per cent	....	....	0.85 to 1.35	0.10	....	....
Vanadium, per cent	....	....	....	....	0.20 to 0.30	0.03

TABLE V.—CHEMICAL COMPOSITION—*Continued.*

Type	Ferritic Steels		Austenitic Steels			
Identification Symbol	B15		B8		B8F[c]	
Grade	Silicon-Chromium-Molybdenum		Stabilized 18 Chromium-8 Nickel (A.I.S.I. Types 321 or 347)		Free Machining 18 Chromium-8 Nickel (A.I.S.I. Type 303)	
	Range	Check Variation, Over or Under[g]	Range	Check Variation, Over or Under[g]	Range	Check Variation Over or Under[g]
Carbon, per cent	0.41 to 0.48	0.02	0.10 max.	0.01 over	0.08 max.	0.01 over
Manganese, per cent	0.45 to 0.65	0.03	2.00 max.	0.04 over	2.00 max.	0.04 over
Phosphorus, max., per cent	0.04	0.005 over	0.04	0.005 over	0.14[d, f]	[a]
Sulfur, max., per cent	0.04	0.005 over	0.03	0.005 over	0.50[b, d, f]	[a]
Silicon, per cent	0.55 to 0.75	0.05	1.00 max.	0.05 over	1.00 max.	0.05 over
Nickel, per cent	....	....	8.00 to 12.00	0.15	8.00 to 10.00	0.10
Chromium, per cent	1.00 to 1.50	0.05	17.00 to 19.00	0.20	17.00 to 19.00	0.20
Molybdenum, per cent	0.40 to 0.60	0.03	....	....	[e]	....
Tungsten, per cent	....	....	....	....	....	....
Vanadium, per cent	....	....	....	....	....	....
Selenium, per cent	....	....	....	....	0.50[b, f]	0.03 over
Titanium, per cent	....	....	5 × carbon content, min.[b]	0.05 over	....	....
Columbium, per cent	....	....	10 × carbon content, min.[b]	0.05 over	....	....
Zirconium, per cent	....	....	....	....	[e]	....

[a] Sulfurized or phosphorized steels are not subject to check analysis, unless misapplication is clearly indicated.
[b] Either element shall be used as specified.
[c] Not recommended for temperatures over 800 F.
[d] If sulfur is added for machinability, phosphorus shall not exceed 0.04 per cent.
[e] Molybdenum, plus zirconium, shall not exceed 0.60 per cent.
[f] Phosphorus and/or sulfur and/or selenium shall total not less than 0.07 per cent.
[g] Unless otherwise specified, separate determinations may vary from the specified ranges, except that elements in any melt must not vary both above and below the specified range.

TABLE V-A.

Grade	Diameter, in.	Minimum Tempering Temperature, deg. Fahr.	Tensile Strength, min., psi.	Yield Point, min., psi.	Elongation in 2 in., min., per cent	Reduction of Area, min., per cent
BA	2½ and under	1200	95 000	70 000	20	50
	Over 2½ to 4, incl.	1200	90 000	65 000	20	50
BB	2½ and under	1100	105 000	80 000	20	50
	Over 2½ to 4, incl.	1100	100 000	75 000	20	50
BC	2½ and under	1000	125 000	105 000	16	50
	Over 2½ to 4, incl.	1000	115 000	95 000	16	45

tence to read as follows by the addition of the italicized words: "These specifications cover *nominal (average) wall* seamless carbon-molybdenum alloy-steel pipe intended for high-temperature service."

Section 5.—Change the required manganese content from the present range of "0.30 to 0.60" to read "0.30 to 0.80" per cent, thus providing a wider range of manganese to bring the composition in line with the A.I.S.I. requirements.

Table I.—Add the same note at the end of this table as recommended above for Table II in Specifications A 106 – 45 T, the formulas to read as follows:

Direction of Test	Formula
Longitudinal	$E = 48t + 15.00$
Transverse	$E = 32t + 10.00$

Section 17.—Add a note at the end of this section to read as follows: "Note.—The minimum wall thickness on inspection is shown in Table II (Appendix III)."

New Table.—Add a new Table II as Appendix III to read as shown in the accompanying Table IV.

Tentative Specifications for Alloy-Steel Castings Suitable for Fusion Welding for High-Temperature Service (A 217 - 44 T):

Section 5.—In Paragraph (*a*) add chemical composition requirements for a new grade WC3 and revise the requirements for the present grade WC4 as follows:

	Grade WC3	Grade WC4 Revised	Grade WC4 Present
Carbon, per cent	0.30[a] max.	0.30[a] max.	0.20 to 0.30
Manganese, per cent	0.70[a] max.	0.70[a] max.	0.40 to 0.70
Phosphorus, max., per cent	0.05	0.05	0.05
Sulfur, max., per cent	0.06	0.06	0.06
Silicon, per cent	0.60 max.	0.60 max.	0.25 to 0.55
Nickel, per cent	......	0.75 to 1.05	0.75 to 1.05
Chromium, per cent	0.40 to 0.70	0.40 to 0.70	0.40 to 0.70
Molybdenum, per cent	0.40 to 0.60	0.40 to 0.60	0.30 to 0.45

[a] In the case of grades WC1, WC2, WC3, and WC4, for each reduction of 0.01 per cent below the specified maximum carbon content, an increase of 0.04 per cent manganese above the specified maximum will be permitted up to a maximum of 1.10 per cent.

In Paragraph (*b*) in the table giving the unspecified alloying elements, add a new grade WC3, with restrictions as follows:

	Grade WC3
Copper, max., per cent	0.25
Nickel, max., per cent	0.50
Tungsten, max., per cent	0.10
Total content of these unspecified elements, max., per cent	1.00

Section 8 (*a*).—Add a new grade WC3 with the following requirements as to tensile properties:

	Grade WC3
Tensile strength, min., psi.	70 000
Yield point, min., psi.	45 000
Elongation in 2 in., min., per cent	22
Reduction of area, min., per cent	35

Tentative Specifications for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 - 44 T):

Section 1.—Change the first sentence in Paragraph (*a*) to read as follows by deleting the words in brackets: "These specifications cover three grades of seamless austenitic chromium-nickel steel still tubes from 2 to $7\frac{1}{2}$ in. in outside diameter and greater than 0.220 in. in minimum [or $\frac{1}{4}$ in. average] wall thickness."

Change the first sentence in Paragraph (*c*) to read as follows by deleting the words in brackets: "In specifying dimensions the purchaser shall specify [either] the outside diameter and minimum wall thickness [or the outside diameter and average wall thickness]."

Table I.—Insert the letters "TP" before the identification symbol numbers 304, 321, and 347.

Tables II, III and IV.—Delete the requirements for average wall thickness from these tables.

Tentative Specifications for Seamless Chromium - Molybdenum Alloy - Steel Pipe for Service at High Temperatures (A 280 - 46 T):

Section 1(*a*).—Revise the first sentence to read as follows by the addition of the italicized words: "These specifications cover *nominal* (*average*) *wall* seamless chromium-molybdenum alloy-steel pipe intended for service at high temperatures in the sizes and wall thicknesses shown in the Appendix."

Table I.—Add the same note to Table I as recommended above for Table II in Specifications A 106 - 45 T, the formulas to read as follows:

Direction of Test	Formula
Longitudinal	$E = 48t + 15.00$
Transverse	$E = 32t + 10.00$

Section 17.—Add a note at the end of this section to read as follows: "The minimum wall thickness on inspection is shown in Table II (Appendix II)."

New Table.—Add a new Table II as Appendix II to read as shown in the accompanying Table IV.

Tentative Specifications for Mild- to Medium - Strength Carbon - Steel Castings for General Application (A 281 - 45 T):

Serial Designation.—Reassign the former A.S.T.M. designation A 27.

Table II.—After the yield point requirement of 35,000 psi. for grade 65–35 insert a reference to footnote "a" and add the following footnote at the bottom of the table:

> When agreed upon by the manufacturer and the purchaser, and when full annealing is required by the purchaser, the yield point value of the 65–35 class shall be 33,000 psi. instead of 35,000 psi.

Tentative Specifications for High - Strength Steel Castings for Structural Purposes (A 282 - 45 T):

Serial Designation.—Reassign the former A.S.T.M. designation A 148.

Table II.—Change the values for reduction of area for grades 120–100 and 150–125 from their present values of "35" and "25", to read "30" and "22" per cent, respectively.

TENTATIVE REVISIONS OF STANDARDS

Standard Specifications for Steel Music Spring Wire (A 228 - 41):

Table I.—In order to present a more rational set of tensile values for the large number of wire sizes covered in these specifications, change the present values for tensile strength to read as prescribed in the accompanying Table VI.

ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that these specifications, revised as follows, be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Specifications for Heat - Treated Carbon - and Alloy - Steel Track Bolts and Nuts (A 183 - 40 T):

Section 4(b).—Change to read as follows:

> (*b*) Carbon and manganese determinations shall be made of each blow of bessemer steel,

TABLE VI.—TENSILE REQUIREMENTS.

Diameter, in.	Present Values		Revised Values	
	Min.	Max.	Min.	Max.
0.004	390 000	440 000	439 000	485 000
0.005	380 000	430 000	426 000	471 000
0.006	370 000	415 000	415 000	459 000
0.007	364 000	409 000	407 000	449 000
0.008	362 000	407 000	399 000	441 000
0.009	360 000	405 000	393 000	434 000
0.010	358 000	398 000	387 000	428 000
0.011	357 000	397 000	382 000	422 000
0.012	356 000	396 000	377 000	417 000
0.013	355 000	395 000	373 000	412 000
0.014	354 000	394 000	369 000	408 000
0.015	353 000	388 000	365 000	404 000
0.016	352 000	387 000	362 000	400 000
0.018	350 000	385 000	356 000	393 000
0.020	348 000	383 000	350 000	387 000
0.022	346 000	381 000	345 000	382 000
0.024	344 000	379 000	341 000	377 000
0.026	342 000	377 000	337 000	373 000
0.028	341 000	376 000	333 000	368 000
0.030	339 000	374 000	330 000	365 000
0.032	337 000	367 000	327 000	361 000
0.034	335 000	365 000	324 000	358 000
0.036	333 000	363 000	321 000	355 000
0.038	332 000	362 000	318 000	352 000
0.040	330 000	360 000	315 000	349 000
0.042	328 000	358 000	313 000	346 000
0.045	325 000	355 000	309 000	342 000
0.048	323 000	353 000	306 000	339 000
0.051	319 000	349 000	303 000	335 000
0.055	315 000	345 000	300 000	331 000
0.059	311 000	341 000	296 000	327 000
0.063	307 000	337 000	293 000	324 000
0.067	303 000	333 000	290 000	321 000
0.072	300 000	330 000	287 000	317 000
0.076	296 000	326 000	284 000	314 000
0.080	293 000	323 000	282 000	312 000
0.085	289 000	319 000	279 000	308 000
0.090	285 000	315 000	276 000	305 000
0.095	282 000	312 000	274 000	303 000
0.100	.....	.....	271 000	300 000
0.102	278 000	308 000	270 000	299 000
0.107	275 000	305 000	268 000	296 000
0.110	.....	.....	267 000	295 000
0.112	273 000	303 000	266 000	294 000
0.121	268 000	298 000	263 000	290 000
0.125	267 000	297 000	261 000	288 000
0.130	265 000	295 000	259 000	286 000
0.135	263 000	293 000	258 000	285 000
0.140	261 000	291 000	256 000	283 000
0.145	259 000	289 000	254 000	281 000
0.150	257 000	287 000	253 000	279 000
0.156	255 000	285 000	251 000	277 000

and determinations for phosphorus and sulfur of each blow of bessemer free-cutting and sulfurized nut steel. For other grades of bessemer steel, determinations for phosphorus and sulfur shall be made at intervals not greater than each ten blows, and the next previous determination may be reported.

Section 14.—Change from its present form to read as follows:

14. The threads on bolts may be rolled or cut. The threads on bolts and nuts shall conform to the American National form of thread as shown on page 12 of the American Standard for Screw Threads, (A.S.A. No.: B1.1–1935). Either a free or wrench turn fit shall be furnished at the option of the purchaser in accordance with the following provisions:

(*a*) *Free Fit.*—The threads of the bolts and nuts shall conform as nearly as practicable to the American Standard limits for tolerance prescribed for the Coarse Thread Series—Free Fit (Class 2) Screws and Coarse Thread Series—Free Fit (Class 2) Nuts.

(*b*) *Wrench Turn Fit.*—The nut shall have a free fit for at least two threads in starting on the bolt and when fully engaged and for the remainder of the screw length shall show the following minimum and maximum resistance to turning as expressed by pounds of pull applied to the end of a 24-in. wrench:

	Resistance, lb. Min.	Resistance, lb. Max.
Regular nuts (Section 3(*b*)) . . .	5	45
High-strength nuts (Section 3(*c*))	5	55

Before packing, nuts shall be screwed on the bolts enough turns to hold them in place until used.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

Standard Specifications for Open-Hearth Carbon-Steel Rails (A 1 – 39):

Section 1.—Change from its present form to read as follows:

1. These specifications cover open-hearth carbon-steel standard Tee rails of nominal weights of 61 lb. per yd. and over. Under special arrangement between the purchaser and the manufacturer, rails of nominal weights of 50 to 60 lb. per yd. may be furnished to the requirements of these specifications for rails of 61 to 69 lb. per yd. except as provided in Section 11(*a*).

Section 3.—In Paragraph (*a*), second sentence, delete the words "dead setting." Omit Paragraph (*b*).

Section 4.—Change from its present form to read as follows by the addition of the italicized words and the omission of those in brackets: "4. A sufficient discard shall be made from each ingot to secure freedom from injurious [piping and undue segregation] *segregation and pipe.*"

Table I.—Add the word "nominal" before the heading "Weight, lb. per yd." Change the present column heading giving weight in pound per yard of 50–69 to read "61–69" with the carbon content to read "0.55 to 0.68 per cent." Change the heading "121–140" to read "121 and over." Change from three groupings of chemistry for rails 70 to 120 lb. per yd., to two groupings with changes in carbon and manganese requirements as follows:

PRESENT REQUIREMENTS

	70–84	85–100	101–120
Carbon, per cent . .	0.53 to 0.70	0.64 to 0.77	0.67 to 0.80
Manganese, per cent	0.60 to 0.90	0.60 to 0.90	0.70 to 1.00

PROPOSED REVISED REQUIREMENTS

	70–90	91–120
Carbon, per cent. .	0.64 to 0.77	0.67 to 0.80
Manganese, per cent	0.60 to 0.90	0.70 to 1.00

Section 6 and Fig. 1.—Delete Paragraph (*b*) from the specifications, and also delete Fig. 1.

Section 7.—Change the first sentence to read as follows by the addition of the italicized words and the omission of those in brackets: "Separate analyses shall be made by the manufacturer from drillings taken from test ingots representing [the second and one of the last full ingots] *one of the first three and one of the last three applied full ingots* of the heat to determine the percentages of carbon and manganese.’

Section 10 (d).—Change this paragraph to read as follows by the addition of the italicized words and the omission of those in brackets: "(*d*) The supports for the test specimens shall be a part of, or firmly secured to, the anvil. These supports shall be spaced 3 ft. between centers for rails under 106 lb. per yd. and [4 ft. for rails over 106 lb. per yd. in weight] *for section 106 lb. to 140 lb. per yd. it shall be 4 ft. For sections over 140 lb. per yd. it shall be 4 ft. 8 in.*

Section 11.—In Paragraph (*a*) under the heading "Weight of Rail, lb. per yd." change the last listing from the present "121 to 140, incl." to read "121 and over."

The first line in the Table giving Height of Drop, ft., for the 50 to 60-lb. rails, incl., is to be placed in parentheses with a footnote reading as follows: "This requirement applies under the arrangement referred to in Section 1."

Section 13.—Reletter as Paragraph (*a*) and add a new Paragraph (*b*) to read as follows:

(*b*) When specified, a progressive all ingot nick and break test with elimination of X-Rays shall be performed as follows:

(*1*) A test specimen representing the top end of the top rail from each ingot of each heat rolled, which has passed the requirements prescribed in Section 11, shall be nicked and broken to determine whether the interior metal is sound. If an interior defect shows on the fracture, the top end of the top rail shall be nicked and broken back. If a fracture free from interior defect is reached at a point permitting the rail to be finished to an acceptable length, the rail and the following rails of the ingot shall be accepted. If not, the rail shall be rejected and a test specimen cut from its bottom end to represent the second rail of the ingot. The second and succeeding rails shall be tested in the same manner.

(*2*) The nick and break tests on the top end of the rails shall be made at increments at the option of the manufacturer and, if necessary, in order to obtain a fracture free from interior defects, the tests shall be carried to the point where the finished rail will be of minimum acceptable length.

(*3*) An interior defect shall be interpreted to mean seams, laminations, cavities, interposed foreign matter, or a distinctly bright or fine-grained structure made visible by the destruction test.

(*4*) Short rails produced under this procedure shall be excluded from consideration in the limiting percentages of Section 15.

Section 15.—Delete this section from the specifications renumbering the subsequent sections accordingly.

Section 20.—Add a new third sentence to Paragraph (*a*) to read as follows: "Auxiliary supports at less than 60 in. may be used for straightening the ends of rails." Also, in the last sentence of this paragraph after "1/32 in." add the phrase "(3/64 in. for 140-lb. rail and over)."

In Paragraph (*b*) change the reference to Paragraph (*a*) to read "Sections 17 and 18 (*a*)."

Change Paragraph (*c*) from its present form to read as follows:

(*c*) When any finished rail shows conditions as described in Section 13 at either end or at any drilled hole, it shall be cut or broken back to sound metal and accepted as a No. 1 or No. 2 short rail subject to the requirement of Sections 15 and 19. If cut back, a fracture specimen shall be furnished the inspector to determine interior condition.

Section 21 (b).—Delete the reference to Section 20 (*c*).

Section 22.—Change the first paragraph from its present form by the omission of the words in brackets to read as follows: "Brands [made so plain and sharp] that [they] may be read as long as the rails are in service shall be rolled on [or hot stamped into] the side of the web of each rail in accordance with the following requirements."

In Paragraph (*a*) change the typical branding from "Weight and Section Number" to read "Weight or Section Number"; delete "O.H." from brand and substitute "C.C." Under "DOE" change "Manufacturer and Mill" to read "Mill Brand."

In Paragraph (*b*) change the first sentence to read as follows by the addition of the italicized words and the omission of those in brackets: "The melt number, *the rail letter*, and the ingot number [as rolled] shall be *hot* stamped in the web of each rail where it will not be covered by the joint bars."

New Section.—Add a new Section 25 to read as follows:

25. *Controlled Cooling.*—(*a*) Rails of a nominal weight of 85 lb. per yd. and over will be control cooled, unless otherwise specified.

(*b*) Rails of a nominal weight of less than 85 lb. per yd. may be control cooled by special arrangement between purchaser and manufacturer.

(*c*) Control cooling shall be in accordance with the following process:[1]

(*1*) All rails shall be cooled in the regular way on hot beds or runways until the temperature is between 1000 and 725 F., then charged immediately into the containers.

(*2*) The temperature of the rails before charging shall be determined with a reliable pyrometer at the top of the rail head at least 12 in. from the end.

(*3*) The handling of rails between the hot bed and the container and during subsequent removal shall be carefully conducted to avoid bending and to minimize cold straightening.

(*4*) The cover shall be placed on the container immediately after completion of the charge and shall remain in place for at least 10 hr. After the removal or raising of the lid of the container no rails shall be removed until the temperature of the top layer of rails has fallen to 300 F. or lower.

(*5*) The temperature between an outside rail and the adjacent rail in the bottom tier of the container, at a point not less than 12 in., nor more than 36 in., from the rail end, shall be recorded with reliable equipment. This temperature shall be the control for judging rate of cooling.

(*6*) The container shall be so protected and insulated that the control temperature shall not drop below 300 F. in 7 hr. for rails 100 lb. per yd. in weight or heavier, from the time that the bottom tier is placed in the container and in 5 hr. for rails of less than 100 lb. per yd. in weight. If for unavoidable mill conditions, this time temperature cycle is not met, the rails shall be considered controlled cooled provided the temperature at a location not less than 12 in. from the end of a rail in the middle tier does not drop below 300 F. in less than 15 hr.

(*7*) The purchaser shall be furnished a complete record of the process for each container of rails on appropriate forms (Note).

NOTE.—The A.R.E.A. Recommended Practice for Controlled Cooling of Railroad Rails refers to a Form 401 D revised in 1946.

(*8*) The letters "C.C." in all brandings shall be removed from all rails failing to meet the above requirements.

(*9*) Rails may be hot stamped in the web ahead of the heat number with the letters "C.H." for control cooled end hardened rails when specified by the purchaser.

Standard Specifications for Low-Carbon Steel Joint Bars (A 3 - 33):

Section 4 (*b*).—Change to read as follows:

(*b*) Carbon and manganese determinations shall be made of each melt of bessemer steel, and determinations for phosphorus and sulfur shall be made at intervals not greater than each ten blows, and the next previous determination may be reported.

Standard Specifications for Steel for Bridges and Buildings (A 7 - 42):

Section 1.—Change from its present form to read as follows:

1. These specifications cover carbon-steel shapes, plates and bars of structural quality for use in the construction of bridges and buildings and for general structural purposes.

NOTE.—The classification by size of flat-rolled carbon-steel, as shown in the Steel Products Manual of the American Iron and Steel Institute is given in the following table (see accompanying Table VII).

Section 2.—Add the words "sheet, strip" to the section title and add a new Paragraph (*d*) to read as follows:

(*d*) Hot-rolled sheets and strip specified to these specifications shall be furnished, unless otherwise specified, to grade C, minimum tensile strength 55,000 psi. as prescribed in the Tentative Specifications for Light Gage Structural Quality Flat Hot-Rolled Carbon Steel (0.2499 and 0.1874 in. to 0.0478 in. in Thickness) (A.S.T.M. Designation: A 245).

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[1] This is the A.R.E.A. Recommended Practice for Controlled Cooling of Railroad Rails, but with some editorial modifications.

Section 9.—Change Paragraph (*b*) to read as follows:

(*b*) Plates $\frac{3}{16}$ in. in thickness, shapes less than 1 sq. in. in cross-section, and bars, other than flats, less than $\frac{1}{2}$ in. in thickness or diameter need not be subjected to tension tests.

TABLE VII.—CLASSIFICATION BY SIZE OF FLAT-ROLLED CARBON STEEL.

Width, in.	Thickness, in.							
	0.2500 and over	0.2499 to 0.2031, incl.	0.2030 to 0.1875, incl.	0.1874 to 0.0568, incl.	0.0567 to 0.0344, incl.	0.0343 to 0.0255, incl.	0.0254 to 0.0142, incl.	0.0141 and under
3½ and under	Bar	Bar	Strip	Strip	Strip	Strip	Sheet	Sheet
Over 3½ to 6, incl.	Bar	Bar	Strip	Strip	Strip	Sheet	Sheet	Sheet
Over 6 to 12, incl.	Plate	Strip	Strip	Strip	Sheet	Sheet	Sheet	Sheet
Over 12 to 32, incl.	Plate	Sheet	Sheet	Sheet	Sheet	Sheet	Sheet	Black plate
Over 32 to 48, incl.	Plate	Sheet	Sheet	Sheet	Sheet	Sheet	Sheet	Sheet
Over 48	Plate	Plate	Plate	Sheet	Sheet	Sheet	Sheet	....

Standard Specifications for Structural Nickel Steel (A 8 – 39):

Section 1.—Change to read as follows:

1. These specifications cover high-strength structural nickel steel shapes, plates, and bars up to $1\frac{1}{2}$ in., inclusive, in thickness, intended primarily for special use in main stress-carrying structural members.

Section 7.—Delete Paragraph (*b*).

Section 11.—Delete Paragraphs (*d*) and (*e*).

Standard Specifications for Wrought Steel Wheels for Electric Railway Service (A 25 – 41):

Section 4.—Add the following as footnote "a" at the end of the table, referenced after "manganese, per cent": "For spun steel wheels the manganese content shall not exceed 1.60 per cent."

Fig. 1.—Revise the lettering on Fig. 1 to read as shown in the accompanying Fig. 1.

Table I.—Delete Table I from the specifications.

Section 8.—Eliminate all reference to wheels operating on T-rails. Also reletter the various paragraphs to agree with the lettering on the revised Fig. 1.

Change the former Paragraphs (*i*) and (*j*) to read as follows, relettering as (*n*) and (*o*) to agree with the new Fig. 1:

(*n*) *Thickness of Plate.*—The dimensions of the plate may vary but shall not be less than that shown in the Table of Standard Designs in Appendix II.

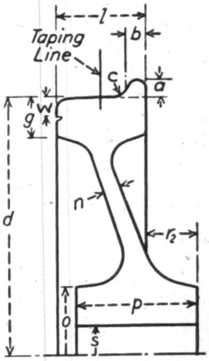


FIG. 1.—Diagram Showing Points at Which the Dimensions Covered by Specifications Are Measured.

For the permissible variations in these dimensions, see Section 8.

The letter used for each dimension in this figure is the same as that of the paragraph of Section 8 on Permissible Variations in Dimensions covering that dimension.

(*o*) *Diameter of Hub.*—The thickness of wall of finished bored hub shall not be less than that shown in the Table of Standard Designs in Appendix II. The outside diameter shall not exceed that specified by more than $\frac{3}{4}$ in. The thickness of the hub wall in any wheel shall not vary more than $\frac{3}{8}$ in. at any two points equidistant from the face of the hub.

Section 9.—Add the following as a new Paragraph (*a*), relettering the remaining paragraphs accordingly: "(*a*) Wheels shall be rough bored and faces of hubs machined. The contour of tread and flange shall be as specified.

Section 10.—Change Paragraph (*a*) from its present form to read as follows:

> (*a*) The date (month and year), name or brand of the manufacturer, manufacturer's serial number and melt number shall be legibly stamped on the back face of the rim approximately $\frac{1}{4}$ in. from the inner edge of the rim. The height of the characters shall not be less than $\frac{5}{8}$ in. for hot stamping nor $\frac{1}{2}$ in. for cold stamping.

Add a new Paragraph (*c*) to read as follows: "(*c*) For spun steel wheels the marking shall be cast in raised letters on the plate."

Appendices.—Add the following as Appendices I and II:

> *Appendix I. Spun Steel Wheels:* Spun steel wheels are usable for electric railway service similar to wrought steel wheels, providing the spun wheels conform to the foregoing specifications for wrought steel wheels.
>
> Spun steel wheels must be manufactured in a manner capable of producing and maintaining high centrifugal fluid compression forces by a high rate of spinning during the entire period in which solidification of metal takes place.
>
> *Appendix II. Wheel Design:* The accompanying Tables 1 and 2 of Dimensions of Standard Steel Wheel Designs, adopted July 30, 1942, by the American Transit Engineering Association, are hereby made a part of Specifications A 25 – 46. The tables are reprinted with permission. (Insert corrected and expanded Tables I and II from EA – 25a)

Standard Specifications for Boiler and Firebox Steel for Locomotives (A 30 – 44):

Section 3.—Change the requirements for manganese for plates $\frac{3}{4}$ in. or under in thickness and plates over $\frac{3}{4}$ in., for the firebox steel, from the present ranges of "0.30 to 0.50" and "0.30 to 0.60" to read "0.30 to 0.80" per cent in both cases.

Table I.—Change the tensile strength range for grade A, firebox, from "52,000 to 62,000" to "55,000 to 65,000" psi.

Standard Specifications for Welded and Seamless Steel Pipe (A 53 – 44):

Section 1.—Revise the second sentence of Paragraph (*a*) to read as follows by the addition of the italicized words: "Pipe ordered under these specifications is *nominal* (*average*) *wall and* is intended for coiling, bending, flanging, and other special purposes, and is suitable for fusion welding."

Number the present note as 1 and add a new Note 2 at the end of this section to read as follows:

> NOTE 2.—When galvanized pipe, as covered by these specifications, is bent or otherwise fabricated to a degree which causes the zinc coating to stretch or compress beyond the limit of elasticity, some flaking of the coating may occur.

Table I.—Add the same note to Table I as recommended above for Table II in Specifications A 106 – 45 T, the formulas to read as follows:

Grade	Formula
A	$E = 56t + 17.50$
B	$E = 48t + 15.00$

Section 13.—Add a note at the end of this section to read as follows: "The minimum wall thickness on inspection is shown in Table VI (Appendix III)."

New Table.—Add a new Table VI as Appendix III to read as shown in the accompanying Table IV.

Standard Specifications for Steel Tie Plates (A 67 – 33):

Section 4 (*b*).—Change to read as follows:

> (*b*) Carbon and manganese determinations (and copper when specified) shall be made of each melt of bessemer steel, and determinations for phosphorus and sulfur shall be at intervals not greater than each ten blows, and the next previous determination may be reported.

Section 9.—Change from its present form to read as follows:

9. The tie plates shall conform to the dimensions specified by the purchaser, subject to the following permissible variations:

(*a*) For plates with shoulders parallel to the direction of rolling, a variation of $\frac{1}{32}$ in. in thickness, $\frac{1}{8}$ in. in rolled width and $\frac{3}{16}$ in. in sheared length will be permitted.

(*b*) For plates with shoulders perpendicular to the direction of rolling, or for plates with rolled crown, a variation of $\frac{1}{32}$ in. in thickness, $\frac{3}{16}$ in. in rolled width and $\frac{1}{4}$ in. in sheared length will be permitted. In the case of plates with shoulders perpendicular to the direction of rolling, the distance from the face of the shoulder to the outside end of the plates shall not vary more than $\frac{1}{8}$ in., and from the face of the shoulder to the inside end not more than $\frac{1}{4}$ in.

(*c*) A variation of 0.025 in. in flatness of the rail seat will be permitted.

(*d*) A variation of $\frac{1}{16}$ in. over the minimum dimension specified for distance between the shoulders of double shoulder tie plates will be permitted.

(*e*) A variation of not more than $\frac{1}{32}$ in. in the location of spike holes and in the length of their sides will be permitted.

Standard Specifications for Low-Carbon Steel Track Bolts and Nuts (A 76 – 33):

Section 4 (*b*).—Change to read as follows:

(*b*) Carbon and manganese determinations shall be made of each melt of bessemer steel, and determinations for phosphorus and sulfur of each melt of bessemer free-cutting and sulfurized nut steel. For other grades of bessemer steel, determinations for phosphorus and sulfur shall be made at intervals not greater than each ten blows, and the next previous determination may be reported.

Standard Specifications for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 – 44):

Section 5.—Change Paragraphs (*a*) and (*b*) from their present form to read as follows, the change entailing the use of a flattening test formula for seamless tubing:

5. (*a*) For lap-welded tubes having wall thicknesses not exceeding 10 per cent of their outside diameters, and providing the thickness does not exceed 0.200 in., a section not less than $2\frac{1}{2}$ in. in length shall stand being flattened between parallel plates, without cracking or showing flaws, until the distance between the plates is five times the wall thickness. For other lap-welded tubes the distance between the plates shall be six times the wall thickness.

(*b*) A section of a seamless tube, not less than $2\frac{1}{2}$ in. in length, shall be flattened cold between parallel plates until the opposite walls of the tube meet. No cracks or breaks in the metal shall occur until the distance between the plates is less than the calculated value of H by the following formula:

$$H = \frac{(1 + e)t}{e + t/D}$$

where:

H = distance between flattening plates in inches,

e = deformation per unit length (constant for a given grade of steel, 0.09 for the low-carbon grade),

t = nominal wall thickness of tube in inches, and

D = actual outside diameter of tube in inches.

Evidence of laminations or burnt material shall not develop during the entire flattening process.

Standard Specifications for Structural Silicon Steel (A 94 – 39):

Section 1.—Change to read as follows:

1. These specifications cover special high-strength structural steel shapes, plates, and bars intended primarily for use in main stress-carrying structural members.

Standard Specifications for Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 105 – 40):

Section 4 (*b*).—Change to read as follows:

(*b*) The material shall be forged by hammering, pressing, rolling, extruding, or upsetting and shall be brought as nearly as practicable to the finished shape and size by hot working and shall be so processed as to cause metal flow during the hot working operation in the direction most favorable for resisting the stresses encountered in service.

Standard Specifications for Structural Steel for Locomotives and Cars (A 113 – 42):

Section 1.—Change to read as follows:

> 1. These specifications cover carbon-steel shapes, plates (except boiler and firebox plates), and bars intended primarily for use in locomotives and car construction.

Section 7.—Change Paragraph (*b*) to read as follows:

> (*b*) Plates $\frac{3}{16}$ in. in thickness, shapes less than 1 sq. in. in cross-section, and bars, other than flats, less than $\frac{1}{2}$ in. in thickness or diameter, need not be subjected to tension tests.

Change Paragraph (*e*) in this section to read as follows by the addition of the italicized figure and the omission of the figure in brackets: "(*e*) For material under $\frac{5}{16}$ in. in thickness or diameter, a deduction from the percentage of elongation in 8 in. specified in Table I of [1.25] *2.00* per cent shall be made for each decrease of $\frac{1}{32}$ in. of the specified thickness or diameter below $\frac{5}{16}$ in."

Standard Specifications for Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 – 44):

Section 1.—Change the second sentence to read as follows by the addition of the italicized words: "Pipe ordered under these specifications is *nominal* (*average*) *wall and is* intended for ordinary uses in steam, water, gas, and air lines, but is not intended for close coiling or bending, or high-temperature service."

In Section 1 number the present note as 1 and add a new Note 2 at the end of this section to read the same as recommended above in Specifications A 53 – 44.

Section 11.—Add a note at the end of this section to read as follows: "The minimum wall thickness on inspection is shown in Table II (Appendix)."

New Table.—Add a new Table III as an Appendix to read as shown in the accompanying Table IV.

Standard Specifications for Structural Steel for Ships (A 131 – 39):

Section 6 (*e*).—Make the same change as recommended above in Section 7 (*e*) of Specifications A 113 – 42.

Standard Specifications for Electric-Resistance-Welded Steel Pipe (A 135 – 44):

Section 1.—Change the first sentence to read as follows by the addition of the italicized words: "These specifications cover two grades of electric-resistance-welded steel pipe 30 in. and under in diameter *with nominal* (*average*) *walls.*"

Table I.—Add the same note at the end of this table as recommended above for Table II in Specifications A 106 – 45 T.

Section 12.—Add a note at the end of this section to read as follows: "The minimum wall thickness on inspection is shown in Table II (Appendix II)."

New Table.—Add a new Table II as Appendix II to read as shown in the accompanying Table IV.

Standard Specifications for Electric-Fusion-Welded Pipe (Sizes 8 in. to but not including 30 in.) (A 139 – 42):

Section 1.—Change the first sentence to read as follows by the addition of the italicized words: "These specifications cover two grades of electric-fusion-welded straight-seam or spiral-seam pipe 4 in. to but not including 30 in. in diameter, with *nominal* (*average*) wall thicknesses up to $\frac{5}{8}$ in., inclusive."

Section 6 (*a*).—Add the same note to the proposed table published as a tentative revision as recommended above for Table II in Specifications A 106 – 45 T.

Section 12.—Add a note at the end of this section to read as follows:

"The minimum wall thickness on inspection is shown in Table II (Appendix II)."

New Table.—Add a new Table II as Appendix II to read as shown in the accompanying Table IV.

Standard Specifications for Seamless Low-Carbon and Carbon-Molybdenum Steel Tubes for Refinery Service (A 161 – 44):

Section 1.—In Paragraph (*a*), sixth line, delete "or $\frac{1}{4}$ in. in average wall."

Change the first sentence of Paragraph (*b*) to read as follows by the deletion of the words in brackets: "In specifying dimensions, the purchaser shall specify [either] outside diameter and minimum wall thickness [or outside diameter and average wall thickness]."

Table I.—Add the same note at the end of this table as recommended above for Table II in Specifications A 106 – 45 T, but substituting "Low-Carbon Steel Tubes" for "Grade A" and "Carbon-Molybdenum Steel Tubes" for "Grade B." Also, insert a new provision covering elongation in 2 in. when the standard round 2-in. gage length test specimen is used with values of 28 per cent for low-carbon tubes and 22 per cent for carbon-molybdenum tubes.

The present values of 35 and 30 per cent, respectively, will remain unchanged covering the basic minimum elongation for walls $\frac{5}{16}$ in. and over in thickness.

Section 4 (a).—Change the manganese content from the present "0.30 to 0.60" to read "0.30 to 0.80" per cent.

Tables II, III and IV.—Delete requirements for average wall thickness.

Explanatory Notes.—Delete explanatory notes.

Standard Specifications for Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 44):

New Table.—Add a new Table II to read as shown in the accompanying Table VIII, renumbering the present Table II as III and the present Table III as IV. Add the same note at the end of this table as recommended above in Table II of Specifications A 106 – 45 T, the formula to read as follows for all types: "E = 40t + 12.50."

TABLE VIII.—TENSILE REQUIREMENTS.

	Ferritic Steel
Tensile strength, min., psi	60 000
Yield point, min., psi	37 000
Elongation in 2 in., min., per cent	30
For longitudinal strip tests a deduction for each $\frac{1}{32}$-in. decrease in wall thickness below $\frac{5}{16}$ in. from the basic minimum elongation of the following percentage	1.50[a]

[a] The following table gives the computed minimum values:

	Elongation in 2 in. min., per cent
Wall Thickness, in.	Ferritic Steel
$\frac{5}{16}$ (0.312)	30.00
$\frac{9}{32}$ (0.281)	28.50
$\frac{1}{4}$ (0.250)	27.00
$\frac{7}{32}$ (0.219)	25.50
$\frac{3}{16}$ (0.188)	24.00
$\frac{5}{32}$ (0.156)	22.50
$\frac{1}{8}$ (0.125)	21.00
$\frac{3}{32}$ (0.094)	19.50
$\frac{1}{16}$ (0.062)	18.00

Standard Specifications for Seamless Cold-Drawn Low-Carbon Steel Heat-Exchanger and Condenser Tubes (A 179 – 44):

Section 1 (b).—Change the first sentence to read as follows by deletion of the words in brackets: "In specifying dimensions, the purchaser shall specify [either] outside diameter and minimum wall thickness [or outside diameter and average wall thickness]."

Section 9.—Change to read as follows by the addition of the note:

9. Each tube shall be tested at the mill and shall withstand a hydrostatic pressure of 1000 psi.

NOTE.—When requested by the purchaser and so stated in the order, tubes shall be tested to one and one-half times the specified working pressure (when one and one-half times the specified working pressure exceeds 1000 psi.) provided the fiber stress corresponding to those test pressures does not exceed 16,000 psi., as determined by the following formula:

$$S = \frac{PD}{2t}$$

where:

P = hydrostatic test pressure in pounds per square inch,

D = outside diameter of the tube in inches, and

t = thickness of the tube wall in inches.

Table I.—Delete the columns referring to inside diameters. Also delete all requirements for average wall tubes.

Explanatory Notes.—Delete the explanatory notes.

Standard Specifications for Forged or Rolled Steel Pipe Flanges for General Service (A 181 - 42):

Section 4 (b).—Change to read as follows:

(*b*) The material shall be forged by hammering, pressing, rolling, extruding, or upsetting and shall be brought as nearly as practicable to the finished shape and size by hot working and shall be so processed as to cause metal flow during the hot working operation in the direction most favorable for resisting the stresses encountered in service.

Standard Specifications for Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 182 - 44):

Section 4 (b).—Change to read the same as recommended above for Section 4 (*b*) of Specifications A 181 - 42.

Standard Specifications for One-Wear and Two-Wear Wrought Steel Wheels (A 186 - 39):

Title.—Change to read "Standard Specifications for One-Wear Wrought Steel Wheels."

Section 1.—Change to read as follows:

1. These specifications cover two classes of one-wear wrought steel wheels for use under cars in freight and industrial service. The two classes are as follows:

Class I, rolled to finish wheels
Class II, machine-finished wheels

Fig. 1.—Revise the lettering on Fig. 1 to read as shown in the accompanying Fig. 2.

Section 9.—In Paragraph (*g*) change the second sentence by deleting the phrase: "for one-wear wheels or 2 in. for two-wear wheels."

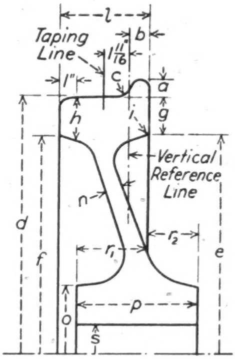


FIG. 2.—Diagram Showing Points at Which the Dimensions Covered by Specifications Are Measured.

For the permissible variations in these dimensions, see Table I or Section 9.

The letter used for each dimension in this figure and in Table I is the same as that of the paragraph of Section 9 on Permissible Variations in Dimensions covering that dimension.

Reletter Paragraph (*g*) as (r_1) and change to read as follows:

(r_1) *Depression of Hub.*—The distance from the back face of rim to the front face of the hub shall not be more but may be $\frac{1}{4}$ in. less than that specified.

Reletter Paragraph (*r*) as (r_2) and change to read as follows:

(r_2) *Projection of Hub.*—The projection of hub beyond the back face of the rim shall not vary more than $\frac{1}{4}$ in. from that specified.

Table I.—Change the present dimension "(*g*)" to "(r_1)" with explanation to read "Depression of hub, distance from back face of rim to front face of rim."

Change the present dimension "(r)" to read "(r_2)" and enter the figure "$\frac{1}{4}$" in both columns for permissible variations in dimensions.

Section 10.—Insert a new Paragraph (*b*) to read as follows, relettering the present Paragraphs (*b*) as (*c*) and (*c*) as (*d*):

(*b*) *Class 1, Rolled to Finish Wheels.*—In class 1 wheels the contour of tread and flange of the wheels may be rolled or machined at the option of the manufacturer.

Class 2, Machine Finished Wheels.—In class 2 wheels the contour of the tread and flange of all wheels furnished must be machined finished.

Standard Specifications for Carbon- and Alloy-Steel Nuts for Bolts for High-Pressure and High-Temperature Service (A 194 – 40):

Section 3 (*c*).—Change the last sentence from its present form to read as follows:

Unless otherwise specified, these classes of nuts shall be quenched and drawn at a temperature not less than that specified, as follows:

Class	Minimum Tempering Temperature, deg. Fahr.
2H	850
3	850
4	1100

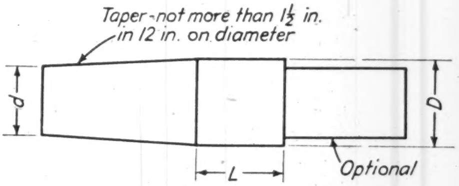
Table II.—Change the title to read "Heat Treatment and Hardness Requirements."

Section 7.—Change the second sentence to read as follows by the addition of the italicized words: "*Except for class 0, a sulfurized steel not subject to check analysis for sulfur,* the phosphorus and sulfur content thus determined shall not exceed that specified in Section 5 by more than 25 per cent, and the other elements shall conform to the specified requirements."

Section 9.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

9. (*a*) Classes 2 H, 3 and 4 nuts, when machined from bar stock, and all other classes of nuts except class 0 shall be capable of meeting the following drift test: A conical mandrel *conforming to the dimensions shown in Fig. 1* (see accompanying Fig. 3) [part of which has a diameter equal to the nominal nut size], shall be forced through the tapped hole to the nominal nut size, cold, without cracking the body of the nut. The test may be continued until the nut is broken for examination of the structure.

(*b*) *Class 0 nuts shall be capable of meeting the drift test specified in Paragraph* (*a*) *except that the mandrel shall be forced through the tapped hole to the pitch diameter of the thread, cold, without cracking the body of the nut.*



D = nominal or pitch diameter, as the case may be.
d = not more than the basic minor diameter of thread.
L = not less than D.

FIG. 3.—Conical Mandrel for Drift Test.

Standard Specifications for Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 – 44):

Section 1 (*c*).—Change the first sentence to read as follows by deletion of the words in brackets: "In specifying dimensions, the purchaser shall specify [either] outside diameter and minimum wall thickness [or outside diameter and average wall thickness]."

Table I.—Change the manganese requirement for grade 2 from the present "0.50 max." to read "0.30 to 0.60" per cent, and change the manganese requirement for grade 3 from the present "0.40 to 0.60" to read "0.30 to 0.60" per cent.

Add three new grades 7, 8, and 9, with chemical requirements to read as follows:

	Grade 7	Grade 8	Grade 9
Carbon, max., per cent	0.15	0.15	0.15
Manganese, per cent	0.30 to 0.60	0.30 to 0.60	0.30 to 0.60
Phosphorus, max., per cent	0.03	0.03	0.03
Sulfur, max., per cent	0.03	0.03	0.03
Silicon, per cent	0.50 to 1.00	0.50 max.	0.50 to 1.00
Chromium, per cent	6.00 to 8.00	4.00 to 6.00	8.00 to 10.00
Molybdenum, per cent	0.45 to 0.65	0.45 to 0.65	0.90 to 1.10

Table II.—Add the same note at the end of this table as recommended above in Table II of Specifications A 106 - 45 T, the elongation formula for all grades to read as follows: "$E = 48\,t + 15.00$."

Section 9 (*b*).—Change from its present form to read as follows: "(*b*) The tubes fabricated from grades 1 to 6, inclusive, and grade ~~8,~~ shall have a Rockwell hardness number not to exceed B85. For tubes fabricated from grades 7 and 9 the maximum Rockwell hardness number shall be B89."

Table III.—Delete the columns referring to inside diameter. Also delete all requirements for average wall tubes.

Explanatory Notes.—Delete the explanatory notes.

Standard Specifications for Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 – 44):

Section 1.—In Paragraph (*a*) delete the words "or $\frac{1}{4}$ in. in average wall" from the seventh and eighth lines.

In Paragraph (*c*) change the first sentence to read as follows by deletion of the words in brackets: "In specifying dimensions, the purchaser shall specify [either] outside diameter and minimum wall thickness [or outside diameter and average wall thickness]."

Table I.—Change the manganese requirement for grade 2 from the present "0.50 max." to read "0.30 to 0.60" per cent.

Add three new grades 7, 8, and 9, the chemical requirements to read the same as above in Table I of Specifications A 199 - 44.

Table II.—Add the same note at the end of this table as recommended above in Table II of Specifications A 106 - 45 T, the elongation formula for all grades to read as follows: "$E = 48\,t + 15.00$." Also, insert a new provision covering elongation in 2 in. when the standard round 2-in. gage length test specimen is used with a value of 22 per cent. The present value of 30 per cent will remain unchanged covering the basic minimum elongation for walls 5/16 in. and over in thickness, longitudinal strip tests, and for all small sizes tested in full section.

Section 9 (*a*).—Change from its present form to read as follows: "(*a*) Tubes fabricated from grades 1 to 6, inclusive, and grade 8 shall have a Brinell hardness number not to exceed 163. For tubes fabricated from grades 7 and 9 the maximum Brinell hardness number shall be 179."

Tables III, IV and V.—Delete all requirements for average wall tubes.

Explanatory Notes.—Delete the explanatory notes.

Standard Specifications for Carbon-Silicon Steel Plates of Ordinary Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels (A 201 - 44):

Section 3.—In Paragraph (*a*) change the reference to "Paragraph (*b*)" to read "Paragraph (*c*)."

Insert a new Paragraph (*b*) to read as follows, relettering the present Paragraph (*b*) as (*c*):

(*b*) The purchaser shall indicate in his orders to the mill whether the rolling mill or the fabricator shall perform the required heat treatment of the plates.

Change the first two sentences in the present Paragraph (*b*) to read as follows by the addition of the italicized words and the omission of those in brackets:

> When a fabricator [who] is equipped to properly perform the work *and* elects to do the required heat treatment *of plates* for grain refinement, [and so indicates in his orders to the mill] he shall accept the plates on the basis of the mill tests. The plate manufacturer shall make the tests prescribed in these specifications, the tension and bend test specimens to be prepared from full thickness pieces heat treated under conditions he considers appropriate for grain refinement and to [give] *meet* the test requirements.

Standard Specifications for Molybdenum-Steel Plates for Boilers and Other Pressure Vessels (A 204 – 44):

Section 3.—Make the same changes as recommended above in Section 3 of Specifications A 201 – 44.

Standard Specifications for Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 209 – 44):

Table I.—Change the manganese requirement for all three grades from the present range of "0.30 to 0.60" to read "0.30 to 0.80" per cent.

Table II.—Add the same note at the end of this table as recommended above in Table II of Specifications A 106 – 45 T, the elongation formula for all grades to read as follows: "$E = 48\,t + 15.00$."

Standard Specifications for Medium-Carbon Seamless Steel Boiler and Superheater Tubes (A 210 – 44):

Table I.—Add the same note at the end of this table as recommended above in Table II of Specifications A 106 – 45 T, the formula to read as follows: "$E = 40\,t + 12.50$."

Standard Specifications for High Tensile Strength Carbon-Silicon Steel Plates for Boilers and Other Pressure Vessels (Plates $4\frac{1}{2}$ in. and Under in Thickness) (A 212 – 44):

Section 3.—Make the same changes as recommended above in Section 3 of Specifications A 201 – 44.

Standard Specifications for Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 – 44):

Table I.—Change the manganese requirement for grades T 3, T 5, T 13, and T 16 from the present "0.50 max." to read "0.30 to 0.60" per cent.

Add two new grades T 7 and T 9, the chemical requirements to read as follows:

Identification Symbol	T 7	T 9
Grade	7 per cent Chromium	9 per cent Chromium
Carbon, max., per cent	0.15	0.15
Manganese, per cent	0.30 to 0.60	0.30 to 0.60
Phosphorus, max., per cent	0.03	0.03
Sulfur, max., per cent	0.03	0.03
Silicon, per cent	0.50 to 1.00	0.50 to 1.00
Chromium, per cent	6.00 to 8.00	8.00 to 10.00
Molybdenum, per cent	0.45 to 0.65	0.90 to 1.10

Table II.—Change the identification symbols T 8, T 18, T 19, and T 20 from their present form to read TP 304, TP 321, TP 347, and TP 316, respectively.

Table III.—Add the same note at the end of this table as recommended above in Table II of Specifications A 106 – 45 T, the elongation formula for the ferritic grade to read "$E = 48\,t + 15.00$"; and for the austenitic grade, "$E = 56\,t + 17.50$."

Section 9 (*c*).—Change from its present form to read as follows: "(*c*) Except for tubes fabricated from ferritic grades T 7 and T 9, the tubes fabricated from ferritic grades shall have a Brinell hardness not to exceed 163 (Rockwell B85). The hardness of the tubes fabricated from austenitic steel shall not exceed a Brinell hardness number of 190 (Rockwell B90).”

Standard Specifications for Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 – 44):

Section 1 (b).—Change the first sentence to read as follows by the deletion of the words in brackets: “Tubes shall be specified to two dimensions only, as follows: Outside diameter and minimum wall thickness [or outside diameter and average wall thickness .”

Table I.—Delete all requirements for average wall tubes.

Explanatory Notes.—Delete the explanatory notes.

Standard Specifications for Manganese-Vanadium Steel Plates for Boilers and Other Pressure Vessels (A 225 – 44):

Section 3.—Make the same changes as recommended above in Section 3 of Specifications A 201 – 44.

Standard Specifications for Carbon-Steel Forgings for General Industrial Use (A 235 – 42):

Delete class D, the 75,000-psi. tensile grade of untreated material from the specifications, making the resulting necessary changes in Section 1 on Scope, Section 7 on Chemical Composition, Table I on Tensile Requirements, and necessary editorial changes in other sections.

Standard Specifications for Carbon-Steel Forgings for Locomotives and Cars (A 236 – 42):

Delete Class B, the 75,000-psi. tensile grade of untreated material from the specifications, making the resulting necessary changes in Section 1 on Scope, Section 7 on Chemical Composition, Table II on Tensile Requirements, and necessary editorial changes in other sections.

Standard Specifications for Alloy-Steel Forgings for Locomotives and Cars (A 238 – 42):

Section 5.—Change from its present form to read as follows:

> 5. (*a*) When boring is specified in the order it shall be performed before normalizing in the case of normalized and tempered forgings; and before quenching, in the case of quenched and tempered forgings.
>
> (*b*) It is recommended that all forgings over 7 in. in diameter be bored. No solid forgings over 10 in. in diameter, and no forging in wall thickness over 8 in. shall be liquid quenched under these specifications.
>
> (*c*) In bored forgings, the diameter of the hole shall be a matter of agreement between the manufacturer and the purchaser. In order to provide adequate quenching, the diameter of the hole shall be not less than 20 per cent of the outside diameter of the forging.
>
> (*d*) Except as specified in Section 17, the position of the rough turning in the sequence of manufacturing operations shall be a matter of agreement between the manufacturer and the purchaser.

Table I.—Change the heading of the first double column under “Size” from the present “Solid Diameter or Thickness, in.” to read “Diameter or Thickness of Solid Forgings, in.,” and change the heading of the second double column from the present “Bored Wall Thickness, in.” to read “Wall Thickness of Bored Forgings, in.”

Standard Specifications for Low-Alloy Structural Steel (A 242 – 42):

Section 1.—Change the first sentence to read as follows by the addition of the italicized words and the omission of those in brackets: “These specifications cover low-alloy structural steel, suitable for welding or riveting, intended primarily for use as [main stress-carrying material of structural members] ***stress-carrying material of structural members where*** *saving in weight and where atmospheric corrosion resistance are important.*"

Section 4.—At the end of Paragraph (*b*) add a new sentence to read as follows: "It is intended that these alloying elements shall be such that the atmospheric corrosion resistance of this steel is materially increased."

TABLE IX.—TENSILE REQUIREMENTS.

	Austenitic Steel
Tensile strength, min., psi	75 000
Yield point, min., psi	30 000
Elongation in 2 in., min., per cent	35
For longitudinal strip tests a deduction for each 1/32-in. decrease in wall thickness below 5/16 in. from the basic minimum elongation of the following percentage	1.75[a]

[a] The following table gives the computed minimum values:

Wall Thickness, in.	Elongation in 2 in., min., per cent
	Austenitic Steel
5/16 (0.312)	35.00
9/32 (0.281)	33.25
1/4 (0.250)	31.50
7/32 (0.219)	29.75
3/16 (0.188)	28.00
5/32 (0.156)	26.25
1/8 (0.125)	24.50
3/32 (0.094)	22.75
1/16 (0.062)	21.00

Standard Specifications for Atomic-Hydrogen-Arc-Welded and Electric-Resistance-Welded Alloy-Steel Boiler and Superheater Tubes (A 249 – 44):

Title.—Change the title to read as follows: "Standard Specifications for Welded Alloy-Steel Boiler and Superheater Tubes," making the necessary editorial corrections in the specifications:

Table I.—Change the identification symbols T 8, T 18, T 19, T 20 and T 24 from their present form to read TP 304, TP 321, TP 347, TP 316 and TP 317, respectively.

Section 6 (*a*).—Revise to read as follows: "(*a*) The material shall conform to the requirements as to tensile properties prescribed in Table II."

Section 19 (*a*).—Change the identification symbols from their present form to read the same as shown above under Table I.

New Table.—Add a new Table II to read as shown in the accompanying Table IX, renumbering the present Table II as III, the present Table III as IV, and the present Table IV as V. Add the same note at the end of this table as recommended above in Table II of Specifications A 106 – 45 T, the formula for all grades to read as follows: "$E = 56t + 17.50$."

TABLE X.—TENSILE REQUIREMENTS.

Identification Symbol	Grade T 1	Grade T 1a	Grade T 1b
Tensile strength, min., psi	55 000	60 000	53 000
Yield point, min., psi	30 000	32 000	28 000
Elongation in 2 in., min., per cent	30[a]	30[a]	30[a]

[a] The following table gives the computed minimum values:

Wall Thickness, in.	Elongation in 2 in., min., per cent
	Grades, T 1, T 1a, T 1b
5/16 (0.312)	30.00
9/32 (0.281)	28.50
1/4 (0.250)	27.00
7/32 (0.219)	25.50
3/16 (0.188)	24.00
5/32 (0.156)	22.50
1/8 (0.125)	21.00
3/32 (0.094)	19.50
1/16 (0.062)	18.00

Standard Specification for Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 – 44):

Table I.—Change the required manganese content for all three grades from the present "0.30 to 0.60" to read "0.30 to 0.80" per cent.

Table II.—Change from its present form to read as shown in the accompanying Table X. Add the same note

at the end of this table as recommended above in Table II of Specifications A 106 - 45 T, the formula for all grades to read as follows: "$E = 48t + 15.00$."

TABLE XI.—TENSILE REQUIREMENTS.

	Grade 1	Grade 2	Grade 3
Tensile strength, min., psi.	50 000	60 000	75 000
Yield point, min., psi.	30 000	35 000	45 000
Basic minimum elongation for walls 5/16 in. and over in thickness:			
Elongation in 8 in., min., per cent	18	14	....
Elongation in 2 in., min., per cent	30	25	20
For walls less than 5/16 in. in thickness a deduction for each 1/32 decrease in wall thickness below 5/16 in. from the basic minimum elongation of the following percentage	1.50[a]	1.25[a]	1.0[a]

[a] The following table gives the computed minimum values:

Wall Thickness, in.	Elongation in 2 in., min., per cent		
	Grade 1	Grade 2	Grade 3
5/16 (0.312)	30.00	25.00	20.00
9/32 (0.281)	28.50	23.75	19.00
1/4 (0.250)	27.00	22.50	18.00
7/32 (0.219)	25.50	21.25	17.00
3/16 (0.188)	24.00	20.00	16.00

NOTE.—The above table gives the calculated minimum elongation values for each 1/32-in decrease in wall thickness. Where the wall thickness lies between two values shown above, the minimum elongation value shall be determined by the following formula:

Grade	Formula
1	$E = 56t + 17.50$
2	$E = 40t + 12.50$
3	$E = 32t + 10.00$

where:

E = elongation in 2 in. in per cent, and

t = actual thickness of specimen, in inches.

Standard Specifications for Welded and Seamless Steel Pipe Piles (A 252 - 44):

Section 1.—Change from its present form to read as follows:

1. These specifications cover nominal (average) wall furnace-welded, electric-welded, and seamless steel black pipe piles of cylindrical shape. They apply to piles in which the steel cylinder acts as a permanent load-carrying member, or alternatively, acts as a shell to form cast-in-place concrete piles. These specifications are not intended to apply to pipe for general structural purposes.

Section 4 (a).—Add a new second sentence to read as follows: "By agreement between the manufacturer and the purchaser, the tension test specimen may be taken from the skelp."

TABLE XII.—STANDARD WEIGHTS AND DIMENSIONS OF WELDED AND SEAMLESS STEEL PIPE PILES.[a]

Outside Diameter, in.	Nominal Thickness, in.	Weight per Lineal Foot (Plain Ends), lb.
8⅝	0.188	16.90
	0.219	19.64
	0.250	22.36
	0.277	24.70
	0.312	27.74
	0.322	28.55
10¾	0.188	21.15
	0.219	24.60
	0.250	28.04
	0.279	31.20
	0.307	34.24
	0.344	38.20
	0.365	40.48
	0.438	48.19
	0.500	54.74
12¾	0.219	29.28
	0.250	33.38
	0.281	37.45
	0.312	41.51
	0.330	43.77
	0.344	45.55
	0.375	49.56
	0.438	57.53
	0.500	65.42
14	0.250	36.71
	0.281	41.21
	0.312	45.68
	0.344	50.14
	0.375	54.57
	0.438	63.37
	0.500	72.09
16	0.250	42.05
	0.281	47.22
	0.312	52.36
	0.344	57.48
	0.375	62.58
	0.438	72.72
	0.500	82.77
18	0.250	47.39
	0.281	53.22
	0.312	59.03
	0.344	64.82
	0.375	70.59
	0.438	82.06
	0.500	93.45
20	0.250	52.73
	0.281	59.23
	0.312	65.71
	0.344	72.16
	0.375	78.60
	0.438	91.41
	0.500	104.13
22	0.312	72.38
	0.375	86.61
	0.438	100.75
	0.500	114.81
24	0.312	79.06
	0.375	94.62
	0.438	110.10
	0.500	125.49

[a] Sizes and weights smaller or greater than those listed may be furnished by special agreement between the manufacturer and the purchaser.

Table I.—Revise Table I to read as shown in the accompanying Table XI.

Table II.—Revise Table II to read as shown in the accompanying Table XII.

Section 8.—Add a note at the end of this section to read as follows: "The minimum wall thickness on inspection is shown in Table III (Appendix)."

New Table.—Add a new Table III as an Appendix to read as shown in the accompanying Table IV.

Standard Specifications for Welded Alloyed Open-Hearth Iron Pipe (A 253 – 44):

Section 1.—Change the second sentence in Paragraph (*a*) to read as follows by the addition of the italicized words: "Pipe ordered under these specifications is *nominal* (*average*) *wall and* intended for coiling, bending, flanging, and other special purposes; also pipe 2 in. and under in nominal diameter may be specified for close coiling."

TABLE XIII.—TENSILE REQUIREMENTS.

	Tensile Requirements
Tensile strength, min., psi	46 000
Yield point, min., psi	30 000
Elongation in 2 in., min., per cent:	
Basic minimum elongation for walls 5/16 in. and over in thickness, longitudinal strip tests, and for all small sizes tested in full section	35
When standard round 2-in. gage length test specimen is used	28
For longitudinal strip tests a deduction for each 1/32-in. decrease in wall thickness below 5/16 in. from the basic minimum elongation of the following percentage	1.75[a]

[a] The following table gives the computed minimum values:

Wall Thickness, in.	Elongation in 2 in., min., per cent
5/16 (0.312)	35.00
9/32 (0.281)	33.25
1/4 (0.250)	31.50
7/32 (0.219)	29.75
3/16 (0.188)	28.00
5/32 (0.156)	26.25
1/8 (0.125)	24.50
3/32 (0.094)	22.75
1/16 (0.062)	21.00

Number the present note as 1 and add a new Note 2 at the end of this section to read the same as recommended above in Specifications A 53 – 44.

New Table.—Insert a new Table I, reading as shown in the accompanying Table XIII, the tensile strength and yield point remaining unchanged. Add the same note at the end of this table as recommended above in Table II of Specifications A 106 – 45 T, the formula for all grades to read as follows: "$E = 56t + 17.50$."

Section 13.—Add a note at the end of this section to read as follows: "The minimum wall thickness on inspection is shown in Table IV (Appendix)."

New Table.—Add a new Table IV as an Appendix to read as shown in the accompanying Table IV.

EDITORIAL CHANGES IN STANDARD AND TENTATIVES

Standard Specifications for Electric-Fusion-Welded Steel Pipe for High-Temperature and High-Pressure Service (A 155 – 42):

Table III.—Revise Table III on permissible overweights of plates ordered to thickness to read as shown in the accompanying Table II.

Tentative Specifications for Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 – 44 T):

Tables I, II and III.—Insert the letters "TP" before the identification symbol numbers 405, 410, 430, 443, 446 and 329.

Section 16.—Insert the letters "TP" before the identification symbol numbers 405 and 410.

Tentative Specification for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 – 44 T):

Table I.—Insert the letters "TP" before the identification symbol numbers 304, 321, 347, 316 and 317.

Section 15.—Insert the letters "TP" before the identification symbol numbers 304, 321 and 347.

Tentative Specifications for Seamless and Welded Austenitic Stainless Steel Tubing for the Dairy and Food Industry (A 270 – 44 T):

Table I.—Insert the letters "TP" before the identification symbol number 304.

REPORT OF COMMITTEE A-3*

ON

CAST IRON

Since the 1944 Annual Meeting of the Society, Committee A-3 on Cast Iron has held one meeting in Pittsburgh, Pa. on February 26, 1946. Another committee meeting will be held during the 1946 Annual Meeting of the Society.

During the past two years, new members elected have exceeded those lost by death or resignation. The total membership of the committee is now 136, of whom 45 are classified as producers, 58 as consumers, and 33 as general interest members. In addition, there are 5 consulting members.

During the past two years, a number of tentative and standard specifications have been revised in order to bring them in line with present practice. New tentative specifications relating to use of cast iron at elevated temperatures were completed late in 1944. In addition, all existing specifications under the jurisdiction of Committee A-3 have been reviewed as to their suitability, and when ncessary have been revised or are in the process of revision. The actions of subcommittees entrusted with such revision, are treated in greater detail later in this report.

Pursuant to the instructions of Committee A-3, the chairman appointed a nominating committee comprising H. Bornstein, T. E. Eagan, and J. S. Vanick to propose nominees for chairman, vice-chairman, and secretary. Each subcommittee has also been instructed to hold an election. The results of the elections of general officers and subcommittee officers were reported at the Annual Meeting, the present officers of the committee having been reelected for the ensuing term of two years.

Subsequent to the 1944 Annual Meeting, Committee A-3 presented to the Society Through the Administrative Committee on Standards several recommendations which are listed in Table I, together with the results of the letter ballot vote in Committee A-3 and the date of acceptance by the Standards Committee. Specifications A 43 appear in the 1945 Supplement to Book of A.S.T.M. Standards, Part I, while the other items listed in Table I appear in the 1944 Book of A.S.T.M. Standards.

TABLE I.—RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS, INCLUDING RECORD OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
Tentative Specifications for:			
Foundry Pig Iron (A 43 - 45 T), *accepted October 30, 1945*......	71	5	19
Automotive Gray Iron Castings (A 159 - 44 T), *accepted November 22, 1944*, as a tentative revision of Standard A 159 - 41.	69	1	20
Lightweight and Thin-Sectioned Gray Iron Castings (A 190 - 44 T) *accepted August 28, 1944*, replacing Standard A 190 - 40 which was accordingly withdrawn..........................	79	1	15
Gray Iron Castings for Pressure-Containing Parts for Temperatures Up to 650 F., (A 278 - 44T) *accepted November 22, 1944*.....	72	0	18

Recommendations Affecting Standards

The committee is submitting revision of one tentative, revision of one standard for immediate adoption and is recommending the adoption of one tentative as standard and the withdrawal of one standard. The standards affected are

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[*] Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

listed in Table I and the recommendations are explained in the reports of the subcommittees directly responsible for them.

These recommendations have been submitted to letter ballot of the committee which consists of 136 members, with the results shown in Table II.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Pig Iron (H. W. Stuart, chairman) prepared new Tentative Specifications for Foundry Pig Iron (A 43–45 T) to replace Standard Specifications A 43–24 which latter standard has been discontinued. The new tentative specifications were approved by Committee A-3 and accepted by the Society, and appear in the 1945 Supplement to Book of A.S.T.M. Standards Part I, p. 171.

TABLE II.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. REVISION OF TENTATIVE			
Def. of Terms Relating to Cast Iron (A 196 - 42 T)	89	0	0
II. ADOPTION OF TENTATIVE AS STANDARD			
Method of Compression Testing of Cast Iron (A 256 - 42 T)	89	0	0
III. REVISION OF STANDARD, IMMEDIATE ADOPTION			
Spec. for Gray Iron Castings (A 48 - 41)	89	0	0
IV. WITHDRAWAL OF STANDARD			
Spec. for Automotive Gray Iron Castings (A 159 - 41)	89	0	0

Subcommittee VI on General Castings (H. Bornstein, chairman).—Tentative Specifications for Lightweight and Thin-Sectioned Castings were prepared, approved by Committee A-3, accepted by the Society and appear in the 1944 Book of A.S.T.M. Standards, Part I under the designation A 190 - 44 T replacing Standard Specifications A 190 - 40 and Emergency Alternate Provisions A 190.

Certain editorial changes were suggested in the Standard Specifications for Gray Iron Castings (A 48 – 41) and unanimously approved at the meeting of Committee A-3 on February 26, 1946. The Committee accordingly recommends the following revisions:

Section 9 (*b*).—Change to read as follows: "If any test specimen shows defective machining or obvious lack of continuity of metal, it shall be discarded and replaced by another specimen."

Section 12.—Change the first sentence to read as follows: "The castings shall conform substantially to the dimensions on drawings furnished by the purchaser or to the dimensions predicated by the pattern supplied by the purchaser, if no drawing has been provided. The castings shall be free from injurious defects."

Explanatory Notes.—Delete Notes 2 and 3 and substitute the following which will be designated as Note 2, renumbering the subsequent notes accordingly: "NOTE 2.—The higher strength irons are usually more expensive to produce and machine."

At the invitation of Subcommittee VI, Mr. MacKenzie has analyzed considerable data to determine the correlation between Brinell hardness and (*1*) tensile strength, (*2*) transverse strength, and (*3*) impact properties. A paper will be presented on this work.

Subcommittee VIII on Cast Iron Culvert Pipe (W. J. Schlick, chairman).—No suggestions for revision of the Standard Specifications for Cast Iron Culvert Pipe (A 142 - 38) have been received, and at the meeting of Committee A-3 on February 26, 1946 it was decided no revision was necessary. However, the chairman is currently canvassing his subcommittee for comments.

Subcommittee X on Nomenclature and Definitions (A. J. Herzig, chairman).—In 1944 this subcommittee recommended the adoption as standard of the Tentative Definitions of Terms Relating to Cast Iron (A 196 – 42 T). This proposal was approved by letter ballot of Committee A-3 on August 24, 1944. Of the 160 committee members, 63 members returned their ballots, of whom 58 members voted affirmatively, 0 negatively, and 5 members marked their ballots "not voting." This action, however, was not presented to the Society in 1945 since Committee A-3 did not present a report to the Society last year.

At a meeting of Subcommittee X on February 25, 1946, it was decided to recommend the following for inclusion in the tentative definitions A 196 – 42 T:

Inoculant.—Inoculants are those materials which, when added to molten cast iron, modify the structure, and thereby change the physical and mechanical properties to a degree not explained on the basis of the change in composition resulting from their use.

Chilled Iron.—Chilled iron is cast iron in which the carbon is retained in the combined form in certain areas sufficient to form a mottled or white structure due to conditions which accelerate cooling to the extent that normal graphitization is prevented in those areas.

Subcommittee X has unanimously approved these definitions and this action of the subcommittee was approved by Committee A-3 at the February 26, 1946, meeting. In view of the inclusion of the above definitions it appears advisable that the definitions remain tentative.

Subcommittee XI on Methods of Testing (V. A. Crosby, chairman) has approved the adoption as standard of the Tentative Method of Compression Testing of Cast Iron (A 256 – 42 T). Tentative Recommended Practice for Torsion Tests of Cast Iron (A 260 – 42 T) is believed to require revision before adoption as standard and has been referred to W. L. Collins, the new chairman of Subcommittee XI.

Subcommittee XV on Impact Testing (J. T. MacKenzie, chairman) has undertaken an investigation of current methods of impact testing to determine if specifications on impact testing are feasible.

Subcommittee XVI on Heat Treatment of Cast Iron (T. E. Eagan, chairman).—At the meeting of Committee A-3 on February 26, 1946, it was voted that this subcommittee should be put on a "stand-by" basis and the subcommittee chairman retained as an adviser on this subject.

Subcommittee XVIII on Automotive Castings (F. J. Walls, chairman).—Tentative Specifications for Automotive Gray Iron Castings (A 159 – 44 T) prepared by this subcommittee, were approved by Committee A-3, accepted by the Society, and appear in the 1944 Book of A.S.T.M. Standards, Part I. These tentative specifications replace the previous Standard A 159 – 41, which the committee accordingly recommends be withdrawn.

Subcommittee XXII on Elevated Temperature Properties of Cast Iron (J. S. Vanick, chairman) in consultation with the American Foundrymen's Association, the War Metallurgy Committee, and the Boiler Code Committee of the American Society of Mechanical Engineers, prepared Tentative Specifications for Gray-Iron Castings for Pressure-Containing Parts for Temperatures up to 650 F. (A 278 - 44 T) which was approved by Committee A-3, accepted by the Society, and appears in the 1944 Book of A.S.T.M. Standards, Part I. It has been decided that this subcommittee be continued to determine whether new specifications can be set for cast irons used at still higher temperatures and largely for nonpressure containing parts.

This report has been submitted to letter ballot of the committee, which consists of 136 members; 89 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. T. MACKENZIE,
Chairman.

E. R. YOUNG,
Vice-Chairman.

C. O. BURGESS,
Secretary.

REPORT OF COMMITTEE A-5*

ON

CORROSION OF IRON AND STEEL

Committee A-5 on Corrosion of Iron and Steel held one meeting during the year, on February 27, 1946, in Pittsburgh, Pa.

Since the last report 4 new members have been elected and 1 member has resigned. The committee membership now totals 131, of whom 57 are classified as producers, 46 as consumers, and 28 as general interest members.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, T. R. Galloway,
First Vice-Chairman, W. F. Markley,
Second Vice-Chairman, L. W. Hopkins.

I. REVISION OF TENTATIVE

The committee recommends the following revision in the Tentative Specifications for Lead Coating (Hot-Dip) on Iron or Steel Hardware (A 267 – 44 T)[1] and its continuation as tentative:

Title and Scope.—Add "alloy" between the words "lead" and "coating."

Section 2.—Change to read as follows by the addition of the italicized words and the omission of the words in brackets:

> 2. The lead that goes into the tank may be any grade of refined commercial lead in pig form conforming to the Standard Specifications for Pig Lead (A.S.T.M. Designation: B 29) *to which tin up to 2.5 per cent has been added*, or it may be secondary metal [or reclaimed lead] (*reclaimed*) containing [not more than] *up to* 2.5 per cent tin. *Where the amount of tin is specified or where the purchase is made on the tin content, then* the tin shall be determined to the nearest 0.1 per cent.

II. WITHDRAWAL OF STANDARD AND ADOPTION OF TENTATIVE AS STANDARD

Since 1938, Committee A-5 has been responsible for two specifications both entitled Specifications for Zinc-Coated (Galvanized) Iron or Steel Sheets: one a standard since 1927 and the other a tentative revision issued in 1938 as tentative specifications.

Specifications A 93 – 38 T[1] have been held as tentative for several years pending attempts to reach agreement with the Federal Specifications Committee on the requirements for coating bend tests. These requirements on bend tests are less severe in the tentative specifications than are the corresponding requirements in the older standard A 93 – 27,[1] which manufacturers state are generally impossible to meet when tin is unavailable, and also not possible to meet with some gages of sheets when tin is available.

The committee recommends that the Standard Specifications A 93 be withdrawn, and that the Tentative Specifications A 93 be adopted as standard with the following revisions:

Section 1.—Delete this section, which is very general in its statement of subject matter covered by the specifications, and replace it by the following which is more informative concerning the classes of zinc coating commercially applied in producing galvanized sheets:

> 1. These specifications cover sheets zinc-coated (galvanized) by the hot-dip process. The weight of coating may be ordered if desired. Galvanized sheets are produced with five classes of zinc coatings, applied by the hot-dip process, as follows:
>
> *Class A.*—Extra heavily coated sheets that are not intended to be formed other than by corrugating.
>
> *Class B.*—Heavily coated sheets that are not

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] 1944 Book of A.S.T.M. Standards, Part I.

intended to be formed other than by corrugating and curving to large radii.

Class C.—Moderately heavily coated sheets for moderate bending.

Class D.—Ordinary coated sheets for general utility. These coatings approximate those of class C except in medium gages in which coatings of class D are appreciably lighter. Class D represents material generally available in warehouse stocks which is not intended for use where relatively long life, represented by classes A, B, and C, or severe forming, represented by class E, is required.

Class E.—Sheets having lighter, more tightly adherent coatings to reduce liability of flaking in severe forming. The sheet maker should be made acquainted with the requirements of fabrication.

Section 6.—Add the following as a new Paragraph (*b*), relettering the subsequent paragraphs accordingly:

(*b*) When coating weights are specified to a class designation, the weight of coating for the gage ordered shall conform to the requirements prescribed in Table II.

Table II.—Add the following as a new Table II (the accompanying Table I), renumbering the subsequent tables accordingly.

TABLE I.—ORDERED WEIGHT OF COATINGS, OUNCE PER SQUARE FOOT

Galvanized Sheet Gage Number	Class A	Class B	Class C	Class D	Class E
8	2.75	2.50	2.00	No Specified Coatings, See Section 1.	No Specified Coatings, See Section 1.
9	2.75	2.50	2.00		
10	2.75	2.50	2.00		
11	2.75	2.50	2.00		
12	2.75	2.50	2.00		
13	2.75	2.50	2.00		
14	2.75	2.50	2.00		
16	2.75	2.50	2.00		
18	2.75	2.50	1.75		
20	2.75	2.50	1.75		
22	2.75	2.50	1.75		
24	...	2.50	1.50		
26	...	2.25	1.25		
27	...	2.00	1.25		
28	...	1.75	1.25		
29	...	1.50	1.25		
30	...	1.25	1.25		

III. Revision of Standard, Immediate Adoption

The committee recommends for immediate adoption the following revision of the Recommended Practice for Safeguarding Against Embrittlement of Hot-Galvanized Structural Steel Products and Procedure for Detecting Embrittlement (A 143 – 35)[1] and accordingly asks for the nine-tenths affirmative vote at the Annual Meeting in order that this revision may be referred to letter ballot of the Society:

Section 5 (*a*).—Change the first sentence to read as follows: "The use of open-hearth steel of tensile properties not higher than that called for in the Standard Specifications for Steel for Bridges and Buildings (A.S.T.M. Designation: A 7) or equivalent, and fabricated in accordance with the recommendations of Section 4 (*b*) and (*c*) should avoid injurious embrittlement."

The recommendation for the above change is based on the uniform opinion of all the available members of the disbanded Subcommittee X of Committee A-5 and others, and is explained as follows by J. J. Shuman, chairman of the currently reconstituted Section on Safeguarding Against Embrittlement, a section of Subcommittee VI of Committee A-5:

> Recommended Practice A 143 was prepared by a former Subcommittee X of Committee A-5, with the cooperation of Samuel Epstein of Battelle Memorial Institute, following a notable investigation of embrittlement of galvanized steel in transmission towers and the like. The recommended practice in its tentative phase (1932) provided for the use of steel to A.S.T.M. Specifications for Structural Steel for Bridges (A 7) when hot-dip galvanizing was intended. At that time Standard A 7 called for a tensile strength of 55,000 to 65,000 psi., but in 1935 it was undergoing revision to a higher tensile range of 60,000 to 72,000 psi.
>
> In advancing A 143 to the status of standard (1935) this change to a higher strength steel was taken into account, and in order to keep the steel grade unchanged the designation A 7 was revised to A 10, a specification for *mild steel plates.* I believe that revision was made as an editorial change which came about from the fact that Subcommittee X had been discharged and the specification was an orphan.

STANDARDS AND TENTATIVE CONTINUED WITHOUT CHANGE

The following six standards have been published for six years or longer without revision. It is recommended that they be continued as standard without change.

Standard Specifications for:

Zinc-Coated (Galvanized) Iron or Steel Tie Wires (A 112 – 33),
Zinc-Coated (Galvanized) Iron or Steel Farm-Field and Railroad Right-of-Way Wire Fencing (A 116 – 39),
Zinc-Coated Iron or Steel Chain-Link Fence Fabric Galvanized After Weaving, (A 117 – 33),
Zinc-Coated (Galvanized) Iron or Steel Barbed Wire (A 121 – 39),
Zinc (Hot-Galvanized) Coatings on Structural Steel Shapes, Plates and Bars, and Their Products (A 123 – 33), and

Standard Methods of Test for:

Weight of Coating on Zinc-Coated Iron or Steel Articles (A 90 – 39).

It is also recommended that the Tentative Specifications for Zinc Coating (Hot-Dip) on Iron and Steel Hardware (A 153 – 42 T) be continued as tentative, as changes under consideration by Subcommittee VI have not yet been satisfactorily formulated.

EDITORIAL CHANGE IN STANDARD

Standard Specifications for Zinc (Hot-Galvanized) Coatings on Structural Steel Shapes, Plates and Bars, and Their Products (A 123 – 33):

Section 5 (a).—Add the following footnote to this section:

[4] See Recommended Practice for Safeguarding Against Embrittlement of Hot-Galvanized Structural Steel Products and Procedure for Detecting Embrittlement (A.S.T.M. Designation: A 143).

CHANGE IN RESPONSIBILITY FOR SPECIFICATION

The committee, following the advice of its Subcommittee VI, recommends that the responsibility for the following specification be transferred to Committee B-8 on Electrodeposited Metallic Coatings:

Tentative Specifications for Electrodeposited Coatings of Cadmium on Steel (A 165 – 40 T). (These specifications cover cadmium coatings in the range of 0.00015 to 0.0005 in. in thickness.)

The recommendations appearing in this report have been submitted to letter ballot of the committee, which consists of 131 members; 84 members returned their ballots, with the results shown in Table II.

TABLE II.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. REVISION OF TENTATIVE			
Spec. for Lead Coating (Hot-Dip) on Iron or Steel Hardware (A 267 – 44 T)	54	0	30
II. ADOPTION OF TENTATIVE AS STANDARD			
Spec. for Zinc-Coated (Galvanized) Iron or Steel Sheets (A 93 – 38 T), as revised	57	0	27
III. REVISION OF STANDARD, IMMEDIATE ADOPTION			
Rec. Practice for Safeguarding Against Embrittlement of Hot-Galvanized Structural Steel Products and Procedure for Detecting Embrittlement (A 143 – 35)	59	0	25
IV. WITHDRAWAL OF STANDARD			
Spec. for Zinc-Coated (Galvanized) Iron or Steel Sheets (A 93 – 27)	49	0	35

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Inspection of Annapolis Tests (Ewart S. Taylerson, chairman).—The report of this subcommittee is appended hereto.

Subcommittee V on Total Immersion Tests (W. J. Jeffries, chairman).—The test program of this subcommittee on underwater exposure tests has been completed. The preparation of a paper covering a statistical study of a large amount of accumulated data is nearing completion.

Subcommittee VI on Specifications for Metallic-Coated Products (T. R. Galloway, chairman).—Substantially all of the information pertaining to specifications presented earlier in this report is the work of Subcommittee VI.

The Section on Galvanized Hardware is studying the need for revisions in the specified weights of coatings on certain classes of rolled, pressed, and forged articles covered in the Tentative Specifications for Zinc Coating (Hot-Dip) on Iron and Steel Hardware (A 153 – 42 T) (class B-2, those less than 3/16 in. in thickness; and class B-3, those less than 8 in. in length).

The Section on Lead-Alloy Coated Sheets has abandoned the project of preparing specifications for lead-alloy coated sheets which was suggested as a war emergency measure. Since this matter is now judged to be no longer urgent, attention is being given to the preparation of specifications for long terne sheets. It is believed that completion of this new project will either lead the way to the formulation of specifications for lead-alloy coated sheets or obviate the need for such specifications.

This subcommittee is investigating the need for the preparation of specifications for galvanized rigid electrical conduit.

Subcommittee VII on Methods of Testing (R. W. Baker, chairman).—The current research of this subcommittee is concerned largely with the evaluation of tests which are now employed, or are proposed, for field use (dropping tests, magnetic tests, and the like) for inspecting specimens to determine the weight or thicknesses of protective coatings. Now that the war is ended, and it is fairly certain that the necessary testing equipment can be purchased, a program is being outlined for further study of the limits of accuracy of pertinent testing methods. The tests made thus far have been made largely by one laboratory with the results outlined below, but testing by additional agencies is needed.

1. Through the use of the magne-gage, magnetic measurement of the thickness of metallic coatings does not appear to be adaptable to hot-dip galvanized sheets because of what is considered to be an unallowable variation of plus or minus 20 per cent from that weight value obtained by standard stripping methods. The variation was found to be less on coated cold-reduced materials, so readings on the uncoated sheets were made. Due to surface irregularities and the way the magnet strikes, a variation of plus or minus 15 per cent was found on bare hot-rolled sheets and a variation of 3 to 7 per cent on bare cold-reduced sheets.
2. Whereas the Hull-Strausser dropping test, like the magnetic test, has been well suited to electroplated coatings, they give an average variation of plus or minus 17 per cent on hot-dip galvanized coatings.

Subcommittee VIII on Field Tests of Metallic Coatings (C. E. Reinhard, chairman).—This subcommittee has carried on the regular inspection of its large collection of galvanized sheet specimens, metallic-coated hardware parts, and wire and wire products which are undergoing atmospheric exposure tests.

This annual report follows the usual precedent of Committee A-5 in choosing to publish detailed inspection records of sheet and hardware specimens only in even-numbered years (as 1946) and to delay until the next succeeding year the printing of detailed records on the performance of wire, fencing, and strand products. The inspection findings on galvanized sheet and metallic-coated hardware are accordingly presented in a more detailed report appended hereto.

Subcommittee VIII has in hand the planning of additional outdoor exposure tests on hardware parts.

This report has been submitted to letter ballot of the committee, which consists of 131 members; 84 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

C. D. HOCKER,
Chairman.

J. B. DIXON,
Secretary.

REPORT OF SUBCOMMITTEE III ON INSPECTION OF ANNAPOLIS TESTS

The specimens of copper-bearing and noncopper-bearing corrugated black sheets that have been exposed at Annapolis, Md., since October 17, 1916, were inspected on April 24, and October 19, 1945. Four failures among the No. 22 gage sheets were found at the April inspection (B-25, K-4, K-6, and K-15), and three at the October inspection (Z-705, I-8, and K-10). No failures of No. 16 gage material were noted this year. A summary of the failures to date is given in Table I.

LOSSES OF WEIGHTS OF SHEETS FROM THE FORT SHERIDAN TEST

It was voted at the meeting of Committee A-5 held in Pittsburgh, Pa., on June 30, 1943, that Subcommittee III obtain and publish further data on the Fort Sheridan, Ill., tests. When this test of Nos. 16 and 22 gage uncoated sheets was discontinued on April 16, 1928, after 11 years' exposure, sample sheets from each group were removed, crated, and stored in the Altoona, Pa., warehouse of the Pennsylvania Railroad. In describing the storage conditions, M. E. McDonnell writes as follows: "While these sheets were stored in Altoona they were in a warm, dry room under lock and key, and any corrosion which could have occurred under these conditions would, in my judgment, be negligible." The sheets were sent to the Vandergrift Plant of the Irvin Works, Carnegie-Illinois Steel Corp. on July 7, 1943. The analysis of each sheet has been published in the *Proceedings* of the Society for 1916 and 1918, and in 1919 the results of umpire analyses for copper content were given.[1]

No. 22 Gage Sheets:

The No. 22 gage sheets were in such condition that it was agreed by the six members of the subcommittee or their representatives present, that the rust could not be removed by pickling without destroying the sheet. Therefore, it was decided to shear a section 2 ft. in width from the center of each sheet. This section was flattened and a specimen approximately 12-in. square sheared and cleaned electrolytically. Using these specimens the losses of weights were calculated, assuming an original weight of 1.25 lb. per sq. ft.

The accompanying Table II shows the record of failures at Fort Sheridan of the No. 22 gage copper-bearing and noncopper-bearing corrugated black sheets for the 11-yr. period of the test, arranged in a manner similar to the present Annapolis test (Table I), as well as the loss of weight per square foot based on that of each 12-in. square specimen after electrolytic cleaning. The results of the Pittsburgh test are shown in Table III, for comparison with Table I (Annapolis) and Table II (Fort Sheridan).

Because there was only about one third of the original weight of the more corroded specimens remaining from the Fort Sheridan test, a large part of the area of each specimen was perforated.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. XVI, Part I, p. 155 (1916); Vol. XVIII, Part I, p. 180 and Plates I and IV (1918); and Vol. XVIX, Part I, p. 181 and Plate III (1919).

TABLE I.—RECORD OF FAILURES AT ANNAPOLIS OF NO. 22 GAGE COPPER-BEARING AND NON-COPPER-BEARING CORRUGATED BLACK SHEETS.
Sheets Exposed October 17, 1916. Inspections Made Semiannually from 1917 to 1946.

Group	Designation	Series	Average Analysis, per cent						Number of Sheets in Test[a]	Life of Each Failed Sheet, yr.																Number of Sheets in Test	Number of Sheets Failed	Average Life of Sheets, yr.	Group
			Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper																					
Z.....	Open-hearth Steel	B	0.032	0.32	0.063	0.042	..	0.019	3	13.5	17.0	19.0	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	16.5	....Z
O.....	Open-hearth Steel	A	0.121	0.536	0.008	0.030	0.249	0.020	12	7.0	9.0	9.0	9.0	9.0	9.5	10.0	10.0	11.0	11.0	13.0	17.0	..	..	..	..	12	12	10.4	....O
B....	Open-hearth Steel	A	0.09	0.38	0.017	0.062	0.036	0.084	3	20.0	20.5	24.0	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	21.5	....B
M....	Open-hearth Steel	B	0.093	0.419	0.013	0.041	0.011	0.185	13	20.0	20.5	23.5	23.5	23.5	24.0	24.0	24.0	24.0	24.0	24.0	24.0	25.0	..	..	..	13	13	23.4	....M
B.....	Open-hearth Steel	A	0.06	0.27	0.014	0.053	0.006	0.187	9	19.5	20.0	21.5	22.0	22.5	23.0	26.0	28.0	28.5	..	..	..	..	..	..	..	9	9	23.4	....B
Z.....	Open-hearth Steel	B	0.067	0.351	0.045	0.052	0.003	0.187	3	27.0	27.5	28.0	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	27.5	....Z
T.....	Open-hearth Steel	B	0.083	0.313	0.009	0.020	0.005	0.227	14	19.5	19.5	19.5	21.5	21.5	21.5	23.0	23.5	23.5	23.5	24.0	24.0	24.0	24.0	..	..	14	14	22.3	....T
K....	Open-hearth Steel (Acid)	A	0.107	0.447	0.091	0.046	0.004	0.237	14	24.5	28.0	28.5	28.5	28.5	29.0	*	*	*	*	*	*	*	*	..	..	14	6	..	....K
H.....	Open-hearth Steel	A	0.069	0.387	0.016	0.027	0.004	0.244	10	19.5	21.0	21.5	23.0	23.5	23.5	23.5	24.0	24.0	25.0	..	..	..	..	..	..	10	10	22.9	....H
Z.....	Open-hearth Steel	B	0.065	0.35	0.050	0.059	0.003	0.264	2	24.5	28.0	..	..	..	..	..	..	..	..	..	..	..	..	..	..	2	2	26.3	....Z
Z.....	Open-hearth Steel	B	0.073	0.42	0.052	0.054	Trace	0.641	3	28.0	29.0	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	2	..	....Z
S......	Open-hearth Iron	B	0.02	0.023	0.006	0.022	0.004	0.022	14	7.0	8.0	8.5	9.0	9.5	10.0	11.0	11.5	11.5	12.5	12.5	12.5	12.5	12.5	..	..	14	14	10.6	.....S
D.....	Open-hearth Iron	A	0.02	0.026	0.004	0.022	0.002	0.024	6	16.0	16.0	16.0	19.5	19.5	23.5	..	..	..	..	..	..	..	..	..	..	6	6	18.4	....D
X.....	Open-hearth Iron	B	0.021	0.055	0.007	0.031	0.004	0.027	14	10.5	13.0	13.5	14.5	14.5	16.0	16.0	16.5	16.5	17.0	17.5	17.5	18.0	19.0	..	..	14	14	15.7	X
C.....	Open-hearth Iron	A	0.01	0.028	0.002	0.027	0.002	0.141	3	17.5	20.5	23.5	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	20.5	C
C.....	Open-hearth Iron	A	0.015	0.028	0.006	0.036	0.003	0.194	9	16.5	16.5	18.5	19.0	19.0	20.0	20.5	21.5	21.5	..	..	..	..	..	..	..	9	9	19.2	C
U.....	Open-hearth Iron	B	0.017	0.074	0.008	0.037	0.005	0.260	13	15.5	16.5	19.0	19.0	19.0	19.0	19.0	19.5	20.0	20.0	21.0	21.0	22.5	..	..	..	13	13	19.3	....U
V.....	Open-hearth Iron	B	0.018	0.051	0.007	0.021	0.005	0.304	14	17.0	17.0	17.5	18.5	18.5	19.0	20.0	20.0	20.5	21.0	22.0	22.0	22.5	24.0	..	..	14	14	20.0	V
A.....	Bessemer Steel	A	0.038	0.386	0.089	0.040	0.007	0.014	15	15.5	16.5	16.5	17.0	17.0	17.0	18.0	19.0	19.0	19.5	19.5	19.5	19.5	19.5	20.0	..	15	15	18.2	....A
Z......	Bessemer Steel	B	0.063	0.41	0.102	0.044	0.009	0.013	3	11.5	11.5	11.5	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	11.5	....Z
Z.....	Bessemer Steel	B	0.077	0.44	0.095	0.036	0.003	0.140	3	27.5	27.5	27.5	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	27.5	....Z
I......	Bessemer Steel	A	0.041	0.365	0.097	0.068	0.008	0.252	15	24.0	26.0	26.5	26.5	26.5	26.5	27.0	28.0	28.0	29.0	*	*	*	*	*	..	15	10	..	I
Z.....	Bessemer Steel	B	0.058	0.37	0.093	0.043	0.009	0.269	3	28.0	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	1	..	....Z
Z.....	Bessemer Steel	B	0.055	0.38	0.110	0.046	..	0.527	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	..	....Z
Y.....	Wrought Iron	B	0.03	0.055	0.139	0.021	0.218	0.020	3	18.0	19.5	23.5	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	20.3	....Y
E.....	Wrought Iron	A	0,033	0.034	0.114	0.021	0.134	0.283	10	19.5	20.5	21.0	21.0	23.0	23.0	23.0	23.0	24.0	25.0	..	..	..	..	..	..	10	10	22.3	....E
							Total		214																	Total....214	195		

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* Indicates unfailed sheet.

[c] The number of sheets in the test indicated in this table is less than the number shown in earlier reports because certain sheets were discarded due to mechanical damage.

TABLE II.—RECORD OF FAILURES AT FORT SHERIDAN OF NO. 22 GAGE COPPER-BEARING AND NONCOPPER-BEARING CORRUGATED BLACK SHEETS.

Sheets Exposed April 9, 1917. Inspections Made Semiannually from 1917 to 1928. Rearrangement of Data from 1928 Report of Committee A-5, Subcommittee III[a].

Group[b]	Designation	Series	Average Analysis, per cent						Number of Sheets in Test	Life of Each Failed Sheet, yr.[e]								
			Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper										
Z 4...........	Open-hearth Steel	B	0.06	0.32	0.065	0.044	0.002	0.018	2	3.1	4.5 A	..			..			
O...........	Open-hearth Steel	A	0.12	0.54	0.008	0.030	0.249	0.020	12	2.7	2.7	2.7	3.1 A	3.1	3.1	3.1	3.1	3.5 B
B 12[c].........	Open-hearth Steel	A	0.02	0.23	0.012	0.055	0.004	0.112	11	11.0	11.0	11.0	11.0	11.0	11.0	11.0	*	*
M...........	Open-hearth Steel	B	0.09	0.42	0.013	0.041	0.011	0.185	14	11.0	11.0 A	*	*	*	*	*	*	*
B 9 and 11....	Open-hearth Steel	A	0.06	0.27	0.014	0.053	0.006	0.156	2	*	*	..	..	..	..	..	..	..
Z 506[d]........	Open-hearth Steel	B	0.06	0.36	0.045	0.053	..	0.174	2	*	*	..	..	..	..	..	..	..
Z 508[d]........	Open-hearth Steel	B	0.06	0.36	0.052	0.064	..	0.247										
T...........	Open-hearth Steel	B	0.08	0.31	0.009	0.020	0.005	0.227	14	11.0	11.0	11.0	11.0	11.0	11.0 A	11.0 B	11.0 C	*
K...........	Open-hearth Steel (acid)	A	0.11	0.45	0.091	0.046	0.004	0.237	16	*	*	*	*	*	*	*	*	*
H...........	Open-hearth Steel	A	0.07	0.39	0.016	0.027	0.004	0.244	11	*	*	*	*	*	*	*	*	*
Z 6	Open-hearth Steel	B	0.06	0.39	0.047	0.047	..	0.286	2	*	*	..	..	..	..	..	..	..
Z 7..........	Open-hearth Steel	B	0.07	0.41	0.042	0.042	..	0.532	2	*	*	..	..	..	..	..	..	..
S............	Open-hearth Iron	B	0.02	0.02	0.006	0.022	0.004	0.022	14	4.5	4.5	4.5	4.5	4.5	5.0	5.0	5.0	5.0
D............	Open-hearth Iron	A	0.02	0.03	0.004	0.022	0.002	0.024	6	5.5	9.4	9.4	10.5 A	10.5 B	11.0 C	..	..	..
X............	Open-hearth Iron	B	0.02	0.06	0.007	0.031	0.004	0.027	14	4.0	5.5	5.5	7.9	8.4	8.4 A	9.0	9.0	9.0
C............	Open-hearth Iron	A	0.01	0.03	0.002	0.027	0.002	0.144	2	11.0	11.0	..	..	..	..	..	..	..
C............	Open-hearth Iron	A	0.02	0.03	0.006	0.036	0.003	0.195	10	8.4 A	9.9 B	10.5 C	11.0	11.0	11.0	11.0	11.0	11.0
U............	Open-hearth Iron	B	0.02	0.07	0.008	0.037	0.005	0.260	14	9.9	9.9	9.9 A	10.5	10.5	10.5	10.5	10.5	11.0 B
V..........	Open-hearth Iron	B	0.02	0.05	0.007	0.021	0.005	0.304	14	10.5	10.5	10.5	11.0	11.0	11.0	11.0	11.0	11.0
A............	Bessemer Steel	A	0.04	0.39	0.089	0.040	0.007	0.014	15	3.1	3.1	3.1	4.0	4.0	4.0	5.5	6.5	7.0 A
Z............	Bessemer Steel	B	0.07	0.44	0.107	0.046	0.003	0.011	2	4.0 A	4.0 B	..	..	..	..	..	..	..
Z 1..........	Bessemer Steel	B	0.06	0.38	0.077	0.035	0.009	0.130	2	*	*	..	..	..	..	..	..	..
I............	Bessemer Steel	A	0.04	0.37	0.097	0.068	0.008	0.252	19	*	*	*	*	*	*	*	*	*
Z 2..........	Bessemer Steel	B	0.06	0.37	0.094	0.038	0.007	0.263	2	*	*	..	..	..	..	..	..	..
Z 3..........	Bessemer Steel	B	0.05	0.37	0.114	0.048	..	0.533	2	*	*	..	..	..	..	..	..	..
Y............	Wrought Iron	B	0.03	0.06	0.139	0.021	0.218	0.020	3	3.5	6.5 A	8.4 B	..	..	..	..	..	..
E............	Wrought Iron.........	A	0.03	0.03	0.114	0.021	0.134	0.283	12	8.4	10.5	*	*	*	*	*	*	*
								Total.....	219									

TABLE II.—RECORD OF FAILURES AT FORT SHERIDAN OF NO. 22 GAGE COPPER-BEARING AND NONCOPPER-BEARING CORRUGATED BLACK SHEETS—*Concluded*

Life of Each Failed Sheet, yr.•										Number of Sheets in Test	Number of Sheets Failed	Average Life of Sheets, yr.	Holes After Cleaning[f]	Loss of Weight, lb. per sq. ft.[e,g]				Group[b]
														A	B	C	Average	
..	..	..	..	..	..	..	..	..	..	2	2	3.8	B+	0.80			0.80	..............Z 4
3.5	3.5	3.5	..	..	..	..	..	..	..	12	12	3.1	B+	0.83	0.84	..	0.84	..............O
•	•	..	..	..	..	..	..	..	..	11	7	..	..	0.70			0.70	............B 12[c]
•	•	•	•	•	..	..	..	..	..	14	2	..	B+	0.72	0.57	0.68	0.66	..............M
..	..	..	..	..	..	..	..	..	..	2	0	..	B+	0.71	0.66	..	0.69	......B 9 and 11
..	..	..	..	..	..	..	..	..	..	2	0	..	B+	0.63	..	..	0.63	..........Z 506[d]
												..	OK	0.48	..	..	0.48	..........Z 508[d]
•	•	•	•	•	..	..	..	..	..	14	8	..	B+	0.65	0.71	0.72	0.69	..............T
•	•	•	•	•	•	•	..	..	..	16	0	..	few	0.46	0.47	0.45	0.46	..............K
•	•	..	..	..	..	..	..	..	..	11	0	..	B+	0.68	0.67	0.72	0.69	..............H
..	..	..	..	..	..	..	..	..	..	2	0	..	OK-few	0.42	0.46	[e]	0.44	..............Z 6
..	..	..	..	..	..	..	..	..	..	2	0	..	OK-1	0.39	0.42	..	0.41	..............Z 7
5.0	5.0	5.0	5.0	5.5	..	..	..	..	..	14	14	4.9	..	..	..	..	..	..............S
..	..	..	..	..	..	..	..	..	..	6	6	9.4	B+	0.82	0.78	0.78	0.79	..............D
9.4	9.9	10.5 B	10.5	10.5	..	..	..	..	..	14	14	8.4	B+	0.84	0.88	[h]	0.86	X
..	..	..	..	..	..	..	..	..	..	2	2	11.0	..	..	..	..	..	C
11.0	..	..	..	..	..	..	..	..	..	10	10	10.6	B+	0.87	0.83	0.80	0.83	C
11.0 C	11.0	11.0	11.0	11.0	..	..	..	..	..	14	14	10.6	B+	0.82	0.81	0.80	0.81	..............U
11.0	11.0	11.0	11.0	11.0	..	..	..	..	..	14	14	10.9	..	..	..	..	..	V
8.4 B	8.4 C	8.4	8.4	8.4	11.0	..	..	..	..	15	15	6.2	B+	0.76	0.77	0.75	0.76	..............A
..	..	..	..	..	..	..	..	..	..	2	2	4.0	B+	0.79	0.77	..	0.78	..............Z
..	..	..	..	..	..	..	..	..	..	2	0	..	B	0.58	0.55	..	0.57	............Z 1
•	•	•	•	•	•	•	•	•	•	19	0	..	OK, B−	0.50	0.51	0.41	0.47	I
..	..	..	..	..	..	..	..	..	..	2	0	..	B−, few	0.52	0.49	..	0.51	............Z 2
..	..	..	..	..	..	..	..	..	..	2	0	..	1–2	0.44	0.43	..	0.44	............Z 3
..	..	..	..	..	..	..	..	..	..	3	3	6.1	B+	0.75	0.72	..	0.74	..............Y
•	•	•	..	..	..	..	..	..	..	12	2	..	B+	0.57	0.59	0.63	0.60	..............E
								Total..........		219	127							

• Indicates unfailed sheets at end of test, April 16, 1928, after exposure of 11 yr.

[a] *Proceedings*, Am. Soc. Testing Mats., Vol. XXVIII, Part I, p. 152 and Plate I (1928).

[b] The suffix number after the Z sheet designations refers to first digit of the three identification numbers

[c] Sheet analysis as given in the Analysis of Steel Sheets, Series "A," for Corrosion Tests, *Proceedings*, Am. Soc. Testing Mats., Vol. XVI, Part I, p. 157 (1916).

[d] Sheet analysis as given in the Analysis of Steel Sheets, Series "B," *Proceedings*, Am. Soc. Testing Mats., Vol. XVIII, Part I, p. 178 and Plates I and IV (1918).

[e] When a "failed" sheet was reserved, its loss of weight can be connected with the "years to failure" by comparing the letter beside "life" with the letter at the top of the loss of weight columns.

[f] Symbols used to designate perforations in specimens 12-in. square:

few	3 to 10 holes.	B	50 to 100 holes.
B−	10 to 50 holes.	B+	Over 100 holes.
		OK	Unperforated.

[g] Original weight assumed to be 1.25 lb. per sq. ft.

[h] Not received.

TABLE III.—RECORD OF FAILURES AT PITTSBURGH OF NO. 22 GAGE COPPER-BEARING AND NONCOPPER-BEARING CORRUGATED BLACK SHEETS.

Sheets Exposed December 12, 1916. Inspections Made Semiannually from 1917 to 1923.
Rearrangement of Data from 1923 Report of Committee A-5, Subcommittee III.[a]

Group	Designation	Series	Average Analysis, per cent						Number of Sheets in Test	Life of Each Failed Sheet, yr.																		Number of Sheets in Test	Number of Sheets Failed	Average Life of Sheets, yr.	Group
			Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper																							
Z....	Open-hearth Steel	B	0.10	0.32	0.069	0.044	0.002	0.018	3	1.8	1.8	1.8	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	1.8	....Z
O....	Open-hearth Steel	A	0.12	0.54	0.008	0.030	0.25	0.020	12	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.3	..	..	..	..	..	..	12	12	1.3	....O
B....	Open-hearth Steel	A	0.09	0.38	0.017	0.062	0.04	0.082	4	1.8	2.3	2.9	3.4	..	..	..	..	..	..	..	..	..	..	..	..	..	..	4	4	2.6	....B
M...	Open-hearth Steel	B	0.09	0.42	0.013	0.041	0.01	0.185	14	3.4	3.4	3.4	3.8	3.8	4.3	4.3	4.3	4.3	4.3	4.3	4.3	4.3	4.8	..	..	..	..	14	14	4.1	....M
B....	Open-hearth Steel	A	0.06	0.27	0.014	0.053	0.006	0.179	9	2.9	3.4	3.8	3.8	4.3	4.3	4.3	4.8	4.8	..	..	..	..	..	..	..	..	..	9	9	4.0	....B
Z....	Open-hearth Steel	B	0.06	0.35	0.049	0.059	..	0.212	3	4.8	4.8	4.8	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	4.8	....Z
T. ..	Open-hearth Steel	B	0.08	0.31	0.009	0.020	0.005	0.227	14	2.9	2.9	3.4	3.4	3.4	3.4	3.4	3.8	3.8	3.8	3.8	3.8	3.8	3.8	..	..	..	..	14	14	3.5	....T
K....	Open-hearth Steel (acid)	A	0.11	0.45	0.091	0.046	0.004	0.237	16	4.8	4.8	5.3	5.3	5.8	5.8	5.8	5.8	5.8	5.8	5.8	*	*	*	*	*	..	..	16	11	..	....K
H....	Open-hearth Steel	A	0.07	0.39	0.016	0.027	0.004	0.244	12	4.3	4.3	4.3	4.3	4.3	4.3	4.3	4.3	4.3	4.8	5.3	5.8	..	..	..	..	..	..	12	12	4.6	....H
Z....	Open-hearth Steel	B	0.07	0.39	0.059	0.058	..	0.268	3	4.8	4.8	4.8	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	4.8	....Z
Z....	Open-hearth Steel	B	0.07	0.41	0.050	0.050	..	0.621	3	4.3	5.3	6.3	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	5.3	....Z
S....	Open-hearth Iron	B	0.02	0.023	0.006	0.022	0.004	0.022	14	1.3	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	..	..	..	..	14	14	1.8	....S
D....	Open-hearth Iron	A	0.02	0.026	0.004	0.022	0.002	0.024	9	1.8	1.8	1.8	2.3	2.3	2.3	2.3	2.3	2.3	..	..	..	..	..	..	..	..	..	9	9	2.1	....D
X....	Open-hearth Iron	B	0.02	0.055	0.007	0.031	0.004	0.027	14	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	..	..	..	..	14	14	1.8	X
C....	Open-hearth Iron	A	0.01	0.028	0.002	0.027	0.002	0.139	2	2.9	3.4	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	2	2	3.2	C
C....	Open-hearth Iron	A	0.02	0.028	0.006	0.036	0.003	0.190	9	2.9	2.9	2.9	2.9	3.4	3.4	3.4	3.8	4.3	..	..	..	..	..	..	..	..	..	9	9	3.3	C
U....	Open-hearth Iron	B	0.02	0.074	0.008	0.037	0.005	0.260	14	2.9	3.4	3.4	3.4	3.8	3.8	3.8	3.8	3.8	3.8	4.3	4.3	4.3	4.3	..	..	..	..	14	14	3.8	....U
V....	Open-hearth Iron	B	0.02	0.051	0.007	0.021	0.005	0.304	14	2.9	2.9	3.4	3.4	3.4	3.8	3.8	3.8	3.8	3.8	3.8	3.8	4.3	4.3	..	..	..	..	14	14	3.7	V
A. ..	Bessemer Steel	A	0.04	0.39	0.089	0.040	0.007	0.014	17	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.8	..	17	17	1.3	....A
Z....	Bessemer Steel	B	0.06	0.37	0.119	0.048	0.003	0.013	3	1.3	1.8	1.8	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	1.6	....Z
Z....	Bessemer Steel	B	0.07	0.40	0.088	0.048	0.009	0.133	3	4.3	5.8	5.8	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	5.3	....Z
I....	Bessemer Steel	A	0.04	0.37	0.097	0.068	0.008	0.252	18	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	18	..	..	I
Z....	Bessemer Steel	B	0 04	0.37	0.084	0.038	0.007	0.257	3	5.3	5.8	5.8	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	5.6	....Z
Z....	Bessemer Steel	B	0.04	0.37	0.100	0.045	..	0.533	2	5.8	5.8	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	2	2	5.8	....Z
Y....	Wrought Iron	B	0.03	0.055	0.139	0.021	0.22	0.020	3	2.3	2.3	2.3	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	2.3	....Y
E....	Wrought Iron	A	0.03	0.034	0.114	0.021	0.13	0.283	12	1.8	1.8	2.3	2.3	2.3	2.3	2.9	2.9	2.9	2.9	3.4	3.4	..	..	..	..	..	..	12	12	2.6	....E
								Total...	230																	Total......		230	207		

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* Indicates unfailed sheets at end of test, March, 1923, after exposure of 6.3 yr.

[a] *Proceedings*, Am. Soc. Testing Mats., Vol. XXIII, Part I, p. 147 and Plate II (1923).

This caused the losses of weights to be less than would have been the case with thicker sheets.

No. 16 Gage Sheets:

The No. 16 gage sheets were cleaned of rust and reweighed in the presence of six members of the subcommittee or their representatives. The heavy rust could not be removed without excessive attack on the base metal, therefore, it was removed mechanically, and the remaining thin film of rust dissolved in muriatic acid with an excess of a good inhibitor present. Table IV gives, in rearranged form, the previously published analyses, perforation data, and losses of weights of the Fort Sheridan sheets, assuming an original weight of 2.50 lb. per sq. ft. For comparison, the records of the No. 16 gage sheets exposed at Pittsburgh are shown in Table V. It is of interest that only three of the No. 16 gage sheets exposed at Annapolis have been declared

TABLE IV.—RECORD OF FAILURES AT FORT SHERIDAN OF NO. 16 GAGE COPPER-BEARING AND NONCOPPER-BEARING CORRUGATED BLACK SHEETS.

Sheets Exposed April 9, 1917. Inspections Made Semiannually from 1917 to 1928.
Rearrangement of Data from 1928 Report of Committee A-5, Subcommittee III.[a]

Group[b]	Designation	Series	Average Analysis, per cent[c]						Number of Sheets in Test	Holes After Cleaning	Loss of Weight, lb. per sq. ft.[e]				Group[b]
			Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper			A	B	C	Average	
ZZ 4	Open-hearth Steel	B	0.02	0.39	0.040	0.028	..	0.030	2	..	0.87	0.83	..	0.85	ZZ 4
OO	Open-hearth Steel	A	0.11	0.37	0.009	0.029	0.007	0.038	12	..	0.70	0.71	0.77	0.73	OO
MM	Open-hearth Steel	B	0.02	0.41	0.010	0.029	0.007	0.057	14	..	0.57	0.55	0.57	0.56	MM
BB	Open-hearth Steel	A	0.06	0.35	0.022	0.048	0.037	0.129	17	..	0.55	0.51	0.63	0.56	BB
ZZ 505	Open-hearth Steel	B	0.02	0.39	0.072	0.081	..	0.268	2	..	0.80	..	..	0.80	ZZ 505
ZZ 506	Open-hearth Steel		0.05	0.37	0.050	0.055	0.009	0.180		..	0.75	..	..	0.75	ZZ 506
TT	Open-hearth Steel	B	0.08	0.31	0.009	0.020	0.006	0.223	14	..	0.75	0.83	0.66	0.75	TT
KK	Open-hearth Steel (acid)	A	0.11	0.43	0.085	0.043	0.005	0.234	19	..	0.53	0.59	0.61	0.58	KK
HH	Open-hearth Steel	A	0.07	0.37	0.011	0.026	0.004	0.237	17	..	0.61	0.69	0.60	0.62	HH
ZZ 7	Open-hearth Steel	B	0.06	0.39	0.049	0.044	..	0.318	2	..	0.67	0.73	..	0.70	ZZ 7
ZZ 6	Open-hearth Steel	B	0.09	0.34	0.038	0.077	0.003	0.641	2	..	0.69	0.73	..	0.71	ZZ 6
SS	Open-hearth Iron	B	0.02	0.02	0.006	0.021	0.006	0.025	12	few	1.10	1.05	1.11	1.09	SS
DD	Open-hearth Iron	A	0.02	0.03	0.004	0.027	0.004	0.028	19	..	0.93	0.95	0.87	0.92	DD
XX	Open-hearth Iron	B	0.02	0.06	0.008	0.030	0.005	0.028	14	..	0.91	1.01	0.98	0.97	XX
CC	Open-hearth Iron	A	0.02	0.03	0.004	0.032	0.003	0.067	12	..	0.81	0.91	0.92	0.88	CC
CCC	Open-hearth Iron	A	0.02	0.03	0.005	0.028	0.005	0.236	12	..	0.89	0.97	0.98	0.95	CCC
UU	Open-hearth Iron	B	0.02	0.07	0.008	0.028	0.005	0.239	13	..	0.81	0.71	0.80	0.77	UU
VV	Open-hearth Iron	B	0.02	0.05	0.007	0.019	0.004	0.299	19	..	0.87	0.65	0.80	0.77	VV
AA	Bessemer Steel	A	0.04	0.42	0.083	0.042	0.004	0.012	17	few[d]	1.06	0.89	0.89	0.95	AA
ZZ	Bessemer Steel	B	0.08	0.45	0.102	0.037	0.008	0.014	2	few	1.12	1.14	..	1.13	ZZ
ZZ 1	Bessemer Steel	B	0.08	0.40	0.091	0.035	0.006	0.154	1	..	0.76	..	..	0.76	ZZ 1
II	Bessemer Steel	A	0.06	0.37	0.095	0.066	0.006	0.247	19	..	0.53	0.51	0.50	0.51	II
ZZ 2	Bessemer Steel	B	0.08	0.34	0.094	0.037	..	0.279	2	..	0.75	0.69	..	0.72	ZZ 2
ZZ 3	Bessemer Steel	B	0.07	0.36	0.082	0.033	..	0.528	2	..	0.56	0.61	..	0.59	ZZ 3
YY	Wrought Iron	B	0.03	0.05	0.123	0.019	0.203	0.023	3	very few	0.94	0.91	0.82	0.89	YY
EE	Wrought Iron	A	0.04	0.03	0.119	0.018	0.125	0.319	12	..	0.62	0.58	0.55	0.58	EE
									Total.. 260						

[a] *Proceedings*, Am. Soc. Testing Mats., Vol. XXVIII, Part I, p. 152 and Plate I (1928).

[b] The suffix number after the ZZ sheet designations refers to first digit of the three identification numbers.

[c] Sheet analyses as given in the Analysis of Steel Sheets, Series "A", for Corrosion Tests, see *Proceedings*, Am. Soc. Testing Mats., Vol. XVI, Part I, p. 155 (1916); Vol. XVIII, Part I, p. 160 and Plates I and IV (1918); and the Analysis of Steel Sheets, Series "B", Vol. XVIII, Part I, p. 163 and Plates I and IV (1918).

[d] The only failures (visible perforations) noted during the 11 yr. of exposure were 4 of the 17 sheets in this group.

[e] Original weight assumed to be 2.50 lb. per sq. ft.

TABLE V.—RECORD OF FAILURES AT PITTSBURGH OF NO. 16 GAGE COPPER-BEARING AND NON COPPER-BEARING CORRUGATED BLACK SHEETS.

Sheets Exposed December 12, 1916. Inspections Made Semiannually from 1917 to 1923.
Rearrangement of Data from 1923 Report of Committee A-5, Subcommittee III.[a]

Group	Designation	Series	Average Analysis, per cent						Number of Sheets in Test	Life of Each Failed Sheet, yr.																			Number of Sheets in Test	Number of Sheets Failed	Average Life of Sheets, yr.	Group
			Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper																								
ZZ.....	Open-hearth Steel	B	0.03	0.40	0.044	0.028	..	0.028	2	5.8	*	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	2	1		.....ZZ
OO ...	Open-hearth Steel	A	0.11	0.37	0.009	0.029	0.007	0.029	12	3.8	4.3	4.3	4.3	4.3	4.8	4.8	4.8	4.8	4.8	4.8	5.8	..	..	..	..	..	..	..	12	12	4.6	.....OO
MM....	Open-hearth Steel	B	0.02	0.41	0.010	0.029	0.007	0.057	14	4.8	4.8	4.8	4.8	4.8	5.3	5.8	5.8	5.8	5.8	5.8	*	*	*	..	..	..	..	..	14	11		.....MM
BB.....	Open-hearth Steel	A	0.06	0.35	0.022	0.048	0.04	0.113	17	4.8	5.3	5.3	5.8	*	*	*	*	*	*	*	*	*	*	*	*	*	..	..	17	4	..	.....BB
ZZ.....	Open-hearth Steel	B	0.06	0.37	0.054	0.064		0.223	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	..	.....ZZ
TT.....	Open-hearth Steel	B	0.08	0.31	0.009	0.020	0.006	0.223	14	*	*	*	*	*	*	*	*	*	*	*	*	*	*	..	..	..	..	..	14	0	..	.....TT
KK.....	Open-hearth Steel (acid)	A	0.11	0.43	0.085	0.043	0.005	0.234	19	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	19	0	..	.....KK
HH.....	Open-hearth Steel	A	0.07	0.37	0.011	0.026	0.005	0.237	15	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	..	..	..	..	15	0	..	.....HH
ZZ.....	Open-hearth Steel	B	0.07	0.40	0.056	0.048		0.364	2	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	2	0	..	.....ZZ
ZZ.....	Open-hearth Steel	B	0.09	0.33	0.032	0.056	0.003	0.575	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	..	.....ZZ
SS.....	Open-hearth Iron	B	0.02	0.02	0.006	0.021	0.006	0.025	14	3.4	3.4	3.4	3.4	3.4	3.4	3.4	3.4	3.8	3.8	3.8	3.8	3.8	3.8	..	..	..	..	..	14	14	3.6	.....SS
XX.....	Open-hearth Iron	B	0.02	0.06	0.008	0.030	0.005	0.028	14	3.4	3.4	3.4	3.4	3.4	3.8	4.3	4.3	4.3	4.3	4.3	4.3	4.8	4.8	..	..	..	..	..	14	14	4.0	XX
DD.....	Open hearth Iron	A	0.02	0.03	0.004	0.027	0.004	0.028	18	4.3	4.3	4.8	4.8	4.8	4.8	4.8	4.8	4.8	4.8	4.8	5.3	5.3	5.3	5.8	5.8	6.3	*	..	18	17	5.0	.....DD
CC.....	Open-hearth Iron	A	0.02	0.03	0.004	0.032	0.003	0.067	12	4.8	5.3	5.3	5.8	5.8	5.8	*	*	*	*	*	*	..	..	..	..	..	..	..	12	6	..	CC
CCC...	Open-hearth Iron	A	0.02	0.03	0.005	0.028	0.005	0.236	12	*	*	*	*	*	*	*	*	*	*	*	*	..	..	..	..	..	..	..	12	0	..	...CCC
UU.....	Open-hearth Iron	B	0.02	0.07	0.008	0.028	0.005	0.239	14	*	*	*	*	*	*	*	*	*	*	*	*	*	*	..	..	..	..	..	14	0	..	.....UU
VV.....	Open-hearth Iron	B	0.02	0.05	0.007	0.019	0.004	0.299	13	*	*	*	*	*	*	*	*	*	*	*	*	*	..	..	..	..	..	..	13	0	..	VV
AA.....	Bessemer Steel	A	0.04	0.42	0.083	0.042	0.004	0.012	17	2.3	2.3	2.3	2.3	2.3	2.3	2.3	2.3	2.3	2.3	2.3	2.3	2.3	2.9	2.9	2.9	2.9	..	..	17	17	2.4	.....AA
ZZ.....	Bessemer Stee	B	0.07	0.40	0.097	0.037	0.008	0.013	3	2.9	2.9	2.9	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	2.9	.....ZZ
ZZ.....	Bessemer Steel	B	0.08	0.40	0.092	0.037	0.004	0.153	1	*	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	1	0	..	.....ZZ
II.....	Bessemer Steel	A	0.06	0.37	0.095	0.066	0.006	0.247	18	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	..	18	0	..	II
ZZ.....	Bessemer Steel	B	0.09	0.31	0.088	0.047	..	0.271	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	..	.....ZZ
ZZ.....	Bessemer Steel	B	0.07	0.36	0.085	0.033	..	0.549	3	*	*	*	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	3	0	..	.....ZZ
YY.....	Wrought Iron	B	0.03	0.05	0.123	0.019	0.20	0.023	3	4.3	4.3	4.8	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	..	3	3	4.5	....YY
EE.....	Wrought Iron	A	0.04	0.03	0.119	0.018	0.13	0.319	12	*	*	*	*	*	*	*	*	*	*	*	*	..	..	..	..	..	..	..	12	0	..	.....EE
								Total...	258																			Total....	258	102		

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* Indicates unfailed sheets at end of test, March, 1923, after exposure of 6.3 yr.

[a] *Proceedings*, Am. Soc. Testing Mats., Vol. XXIII, Part I, p. 147 and Plate II (1923).

failures due to perforations during the 29-yr. period of exposure. These were from group "A" (low-copper Bessemer steel) which had the shortest average life at Pittsburgh and was the only No. 16 gage material to show perforations at Fort Sheridan.

This report of Subcommittee III has been unaminously approved by the subcommittee.

Respectfully submitted on behalf of the subcommittee,

EWART S. TAYLERSON,
Chairman.

REPORT OF SUBCOMMITTEE VIII ON FIELD TESTS OF METALLIC COATINGS

The regular inspections of the exposure tests of specimens of galvanized sheet, metallic-coated hardware, wire, and wire products were continued during the year. One inspection of the Key West, Fla., specimens was made in April, 1946. The present report comprises largely the detailed data obtained in the sheet and hardware tests.

GALVANIZED SHEET EXPOSURE TESTS

A summary of the failures of coatings and sheets (perforations) at each of the test locations has been assembled in Table I. For each location are shown the number of specimens of each weight of coating class on test, the number of specimens showing coating failure between October, 1943, and October, 1945, and, where sufficient failures have occurred, the average minimum and maximum life of the coating to first rust, the average time to complete rusting, and a record of the perforations noted between October, 1943, and October, 1945.

Since the tests at Altoona and Brunot Island are nearing completion, a detailed record of the perforation of individual sheets can be given. These records will be found in Tables II and III, together with the chemical composition of the iron or steel. The data for uncoated and coated No. 22 gage sheets only are included, providing a direct comparison for sheets of this gage.

A brief résumé of the status of the tests at each location follows. The exposure period is approximately 19½ yr. for all locations.

Altoona, Pa.:

Coating failure and complete removal of coating have occurred on all originally coated specimens. All uncoated specimens had previously perforated and perforations had occurred in the coated sheets in all weights of coating classes.

The difference between the life of copper-bearing and noncopper-bearing sheets is marked whether originally uncoated or coated.

Brunot Island (Pittsburgh), Pa.:

Coating failures and complete removal of coating have occurred on all originally coated specimens. All uncoated specimens had previously perforated. Some perforations have now occurred in all weights of coating classes of the originally coated sheets.

At this location the difference between the life of copper-bearing and noncopper-bearing sheets is as marked as at Altoona.

Sandy Hook, N. J.:

Coating failure has now occurred on all originally coated sheets. Complete coating removal is confined to the 1.50-oz. per sq. ft. weight and under coating classes. No originally coated sheets have perforated. All uncoated sheets had been reported perforated in earlier reports.

State College, Pa.:

During the past two years only three additional coating failures were observed at this location. The coating failures are confined to the 2.0-oz. per sq. ft.

weight and under coating classes. No perforations have been recorded in either the uncoated or coated groups.

Key West, Fla.:

Some additional sheets showed the perforation from corrosion originating on the under side at the April, 1946, inspection. Some sheets in all weights of coating classes now show this condition.

Normal coating failure (rusting) is still confined to the 0.75-oz. per sq. ft. coated sheets and the areas rusted continue to be small. A few sheets now show rusting in the areas previously spotted by corrosion from the under side, making it difficult to distinguish the two types of failure except in those instances where visible perforation has occurred.

All uncoated sheets had previously perforated. The difference between the life of copper-bearing and noncopper-bearing sheets was not as marked at this location as at the northern severe industrial locations.

BEND TESTS

The inspection of the numerous bend specimens is being continued at all locations. No summarization of the available data has been made. The main purpose of the inclusion of this item in the report is to serve notice that these tests are being continued even though no mention has been made in recent reports. A general statement can be made that bending has not seriously decreased the useful life of the coatings or the sheets. In addition, spots at which the coatings flaked due to the bending have not become points of failure at any test location. Where general rusting has developed, the originally flaked spots are no longer distinguishable from the originally unflaked areas. At Key West only a few of the largest flaked areas on heavily coated sheets show small rust areas and all specimens carrying 0.75 oz. per sq. ft. of coating show pronounced rusting. The rusting of the bend specimens indicates a slightly greater rate of corrosion than on corresponding sheet specimens, which may be due as much to the exposure position as to the bending.

EXPOSURE TESTS OF METALLIC-COATED HARDWARE

The data obtained in the routine semiannual inspection of the hardware specimens at the three remaining test locations have been assembled in Table IV. These cover the period from April, 1943, to October, 1945. The Key West exposures were inspected only twice: April, 1944, and April, 1946. The test racks at this location were rebuilt during January, 1946. All of the test racks have now been rebuilt. The arrangement of the specimens on the reconstructed Key West rack is in accordance with the original plan since relatively few specimens had 100 per cent of their surface area rusted.

A brief résumé of the status of the tests at each location follows.

Altoona, Pa.:

All tests discontinued in 1942. Last tabulation of data was in 1944 report.[1]

Brunot Island (Pittsburgh), Pa.:

All tests discontinued, specimens were stolen during the summer of 1939. Last tabulation of data was in 1944 report.[1]

State College, Pa.:

Only zinc, aluminum, and lead-coated specimens remain on test at this location. A few of the hot-dip zinc-coated specimens show yellow alloy layer rust but remain free from rough red rust. Some

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[1] Report of Subcommittee VIII on Field Tests of Metallic Coatings, Committee A-5, *Proceedings*, Am. Soc. Testing Mats., Vol. 44, p. 92 (1944).

TABLE I.—RECORD OF COATING FAILURES AND PERFORATIONS.

Description		Number of Specimens Rusted						Life of Coating to First Rust, yr.			Average Time to Complete Coating Removal, yr. (100R)	Number of Specimens, Perforated[f]				
Gage	Weight of Coating, oz. per sq. ft.	In Test	Oct., 1943	April, 1944	Oct., 1944	April, 1945	Oct., 1945	Average	Minimum	Maximum		Oct., 1943	April, 1944	Oct., 1944	April, 1945	Oct., 1945
ALTOONA, PA. (EXPOSED APRIL 22, 1926)[a]																
No. 16	2.5	10	10	...	...	...	...	4.4	4.3	4.8	11.1	2	2	2	2	2
No. 22	2.5	18	18	...	...	...	...	4.6	3.3	6.3	11.2	3	3	3	3	3
No. 22	2.0	18	18	...	...	...	...	3.8	3.3	4.3	8.1	3	3	4	7	8
No. 22	1.5	18	18	...	...	...	...	3.0	2.3	3.3	5.7	10	10	11	14	14
No. 22	1.25	18	18	...	...	...	...	2.4	2.3	3.3	4.3	12 (1)[b]	12	12	14	15
No. 22	0.75	14	14	...	...	...	...	1.9	1.4	2.3	3.1	11 (1)[b]	11	11	11	11
No. 28	0.75	10	10	...	...	...	...	2.1	1.4	2.3	3.1	10 (1)[b]	...	...	...	...
No. 22	Uncoated	10	...	...	...	...	...	...	...	...	...	10 (1)[b]	...	...	...	...
BRUNOT ISLAND, PA.[c] (EXPOSED APRIL 21, 1926)																
No. 16	2.5	12	12	...	...	...	...	5.2	4.3	5.8	14.2	2	2	2	2	2
No. 22	2.5	18	18	...	...	...	...	5.9	4.0	8.5	14.3	0	0	0	1	3
No. 22	2.0	18	18	...	...	...	...	4.6	3.5	5.8	11.7	2	2	3	3	3
No. 22	1.5	18	18	...	...	...	...	3.5	3.3	4.3	8.6	5	5	8	9	9
No. 22	1.25	18	18	...	...	...	...	3.2	2.5	3.3	6.1	6	6	7	8	10
No. 22	0.75	14	14	...	...	...	...	2.2	2.0	2.3	4.2	4	6	6	7	8
No. 28	0.75	10	10	...	...	...	...	2.2	2.0	2.3	3.8	8	8	8	9	9
No. 22	Uncoated	10	10	...	...	...	...	...	...	...	..	10	..	...	...	...
SANDY HOOK, N. J. (EXPOSED MAY 20, 1926)																
No. 16	2.5	12	12	...	...	...	..	11.8	10.4	13.4	...	0	0	0	0	0
No. 22	2.5	18	17	17	18	...	...	13.1	10.4	18.4	...	0	0	0	0	0
No. 22	2.0	18	18	...	...	...	...	10.0	8.5	12.4	...	0	0	0	0	0
No. 22	1.5	18	18	...	...	...	...	7.6	6.4	9.5	17.9[d]	0	0	0	0	0
No. 22	1.25	18	18	...	...	...	...	6.8	5.9	7.4	15.2	0	0	0	0	0
No. 22	0.75	14	14	...	...	...	...	4.8	4.4	6.4	11.3	0	0	0	0	0
No. 28	0.75	10	10	...	...	...	...	4.9	4.4	6.4	11.0	0	0	0	0	0
No. 22	Uncoated	10	...	...	...	...	...	...	...	...	...	10	...	...	...	...
STATE COLLEGE, PA. (EXPOSED APRIL 23, 1926)																
No. 16	2.5	12	0	0	0	0	0	...	...	...	...	0	0	0	0	0
No. 22	2.5	18	0	0	0	0	0	...	...	...	...	0	0	0	0	0
No. 22	2.0	18	1	1	2	2	2	...	17.5	...	...	0	0	0	0	0
No. 22	1.5	18	13	13	15	15	15	17.0[d]	14.0	...	...	0	0	0	0	0
No. 22	1.25	18	18	...	...	...	...	14.6	12.0	16.5	...	0	0	0	0	0
No. 22	0.75	14	14	...	...	...	...	10.0	6.5	11.5	...	0	0	0	0	0
No. 28	0.75	10	10	...	...	...	...	11.2	9.5	13.0	...	0	0	0	0	0
No. 22	Uncoated	10	...	...	...	...	...	...	...	...	...	0	0	0	0	0
KEY WEST, FLA.[f] (EXPOSED JUNE 9, 1926)																
							April, 1946									April, 1946
No. 16	2.5	12	...	0	...	...	0	...	...	...	...	...	(2) 1	...	...	(6) 2
No. 22	2.5	18	...	0	...	...	0	...	...	...	...	...	(0) 0	...	...	(2) 0
No. 22	2.0	18[e]	...	0	...	...	0	...	...	...	...	...	(6) 3	...	...	(10) 5
No. 22	1.5	18	...	0	...	...	0	...	...	...	...	...	(13) 8	...	...	(16) 9
No. 22	1.25	18[e]	...	0	...	...	0	...	...	...	...	...	(16) 13	...	...	(17) 13
No. 22	0.75	14	...	8	...	...	14	17.5	13.8	19.8	...	...	(13) 12	...	...	(14) 11
No. 28	0.75	10	...	7	...	...	9	17.6[d]	14.8	...	...	...	(9) 3	...	...	(9) 3
No. 22	Uncoated	10	...	...	...	...	...	...	...	...	...	...	10	...	...	10

[a] A detailed record of coating failures may be found in the 1933 Report, Table I; see *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 150 (1933).

[b] Number of sheets blown away by storm which had not previously failed are indicated in parentheses. They are included in the total but not in the averages.

[c] A detailed record of coating failures may be found in the 1938 Report, Table II; see *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part I, p. 86 (1938). Uncoated sheet failures are given in the 1940 Report, Table VI; see *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 110 (1940).

[d] Values so marked are not final; not all specimens in this particular group have failed.

[e] Two sheets in these groups removed after 14.8 yr. All showed the characteristic perforation except one in the 2.0-oz. class.

[f] At Key West, perforations are indicated by a yellow-brown stain showing through the coating on the upper surface from corrosion originating on the under side of the sheet. The sheet may be neither visibly perforated nor show normal coating failure on the top surface. Two values are shown for perforation at this location. Those values in parentheses refer to the stain spots described and correspond to the values given in previous reports. It should be remembered that while the other values for this location (those not in parentheses) refer to visible perforations, they develop from the stain spot or indicated perforations and cannot be compared with the values for normal perforations at other locations.

TABLE II.—YEARS TO PERFORATION OF UNCOATED AND COATED STEELS AT ALTOONA, PA.

Identification	Class of Material	Average Chemical Composition, per cent									Years to Perforation[a]										
		Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper	Nickel	Chromium	Molybdenum	Uncoated	Galvanized Coating Class, oz. per sq. ft.									
												0.75 to 1.0		1.25		1.50		2.0		2.5	
R........	Open-hearth iron	0.011	0.021	0.004	0.018	0.004	0.032	0.023	[d]	0.004[b]	4.27	[e]	[e]	14.5	16.0	14.5	17.0	18.5	16.5	*	*
V........	Open-hearth iron	0.018	0.053	0.005	0.032	0.004	0.051	0.045	[d]	0.007[b]	4.27	9.0	13.0	13.5	15.0	14.5	17.0	17.5	19.0	[f]	17.5
U........	Open-hearth iron	0.015	0.020	0.004	0.024	0.005	0.087	0.106	[d]	0.009[b]	4.27	[e]	[e]	[e]	[e]	[e]	[e]	[e]	[e]	[e]	[e]
N........	Open-hearth iron	0.013	0.057	0.007	0.029	0.003	0.167	0.057	[d]	0.004[b]	7.51	13.0	15.0	15.0	[f]	17.0	19.0	*	*	*	*
T........	Open-hearth iron	0.016	0.157	0.007	0.028	0.004	0.228	0.073	[d]	0.004[b]	8.52	13.5	15.0	15.0	17.0	18.5	17.0	*	*	*	*
Y........	Open-hearth steel	0.028	0.346	0.067	0.050	0.005	0.029	0.003[b]	0.013	[c]	2.03	4.3	8.5	14.0	15.0	14.0	17.0	17.5	19.0	15.5	17.5
S........	Open-hearth steel	0.040	0.488	0.072	0.041	0.003	0.051	0.006[b]	0.094	[c]	9.00	13.5	15.0	17.0	19.0	19.0	*	*	*	*	*
C........	Open-hearth steel	0.066	0.458	0.053	0.045	0.003	0.222	0.003[b]	0.000[b]	[c]	[f]	[f]	[f]	19.0	19.5	*	19.0	*	*	*	*
K........	Open-hearth steel	0.023	0.460	0.066	0.045	0.003	0.268	0.023[b]	0.031	[c]	13.47	*	*	*	*	*	*	*	*	*	*
W........	Wrought iron	0.021	0.048	0.143	0.019	0.210	0.025	0.020[b]	[d]	0.003[b]	5.00	[e]	[e]	14.5	15.0	15.0	17.0	19.0	19.5	*	*

* Sheet not perforated.

[a] Two values shown are for top and bottom sheets.

[b] Average of determinations in one laboratory.

[c] Less than 0.003 per cent; results of one laboratory.

[d] Less than 0.006 per cent; results of one laboratory.

[e] No sheet in test.

[f] Sheet lost due to breaking of straps.

TABLE III.—YEARS TO PERFORATION OF UNCOATED AND COATED STEELS AT BRUNOT ISLAND (PITTSBURGH), PA.

Identification	Class of Material	Average Chemical Composition, per cent									Years to Perforation[a]										
		Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper	Nickel	Chromium	Molybdenum	Uncoated	Galvanized Coating Class, oz. per sq. ft.									
												0.75 to 1.0		1.25		1.5		2.0		2.5	
R........	Open-hearth iron	0.011	0.021	0.004	0.018	0.004	0.032	0.023	[d]	0.004[b]	2.03	[e]	[e]	15.5	17.0	17.5	17.5	*	18.5	19.5	*
V........	Open-hearth iron	0.018	0.053	0.005	0.032	0.004	0.051	0.045	[d]	0.007[b]	2.27	15.4	13.5	17.5	18.5	18.5	18.5	*	*	19.5	*
U........	Open-hearth iron	0.015	0.020	0.004	0.024	0.005	0.087	0.106	[d]	0.009[b]	2.53	[e]	[e]	[e]	[e]	[e]	[e]	[e]	[e]	[e]	[e]
N........	Open-hearth iron	0.013	0.057	0.007	0.029	0.003	0.167	0.057	[d]	0.004[b]	3.00	16.5	18.0	19.0	19.5	19.5	*	*	*	*	*
T........	Open-hearth iron	0.016	0.157	0.007	0.028	0.004	0.228	0.073	[d]	0.004[b]	9.00	19.0	19.5	*	*	*	*	*	*	*	*
Y........	Open-hearth steel	0.028	0.346	0.067	0.050	0.005	0.029	0.003[b]	0.013	[c]	2.03	*	*	15.5	13.5	15.5	17.5	17.0	14.0	19.0	*
S........	Open-hearth steel	0.040	0.488	0.072	0.041	0.003	0.051	0.006[b]	0.094	[c]	3.25	14.4	18.0	*	*	19.0	*	*	*	*	*
C........	Open-hearth steel	0.066	0.458	0.053	0.045	0.003	0.222	0.003[b]	0.000[b]	[c]	12.48	*	*	*	*	*	*	*	*	*	*
K........	Open-hearth steel	0.023	0.460	0.066	0.045	0.003	0.268	0.023[b]	0.031	[c]	16.5	*	*	*	*	*	*	*	*	*	*
W........	Wrought iron	0.021	0.048	0.143	0.019	0.210	0.025	0.020[b]	[d]	0.003[b]	3.00	[e]	[e]	17.5	19.5	18.0	13.0	*	*	*	*

* Sheet not perforated.

[a] Two values shown are for top and bottom sheets.

[b] Average of determinations in one laboratory.

[c] Less than 0.003 per cent; results of one laboratory.

[d] Less than 0.006 per cent; results of one laboratory

[e] No sheet in test.

TABLE IV.—REPORT ON INSPECTIONS OF METALLIC COATINGS ON HARDWARE, STRUCTURAL SHAPES, TUBULAR GOODS, ETC., EXPOSED TO ATMOSPHERE ON TEST RACKS AT FIVE LOCATIONS.

Abbreviations and symbols used: 0, no rust; 5, 10, 25, 100, approximate percentages; R, rusting of base metal; Y, yellow or orange yellow rust; *, rust spots widely distributed; D, black or dark surface film (soot or dirt); a, in angles; <, less than; e, edges; TH, threads; TR, trace; SL, slight; N, near; S, stain; X, 100 per cent rust listed in previous reports.

Coating	Weight, oz. per sq. ft. of surface	Exposure at State College, Pa.						Exposure at Sandy Hook, N. J.						Exposure at Key West, Fla.	
		4–29–43 736 Weeks	10–13–43 758 Weeks	4–27–44 785 Weeks	10–10–44 808 Weeks	4–26–45 835 Weeks	10–17–45 858 Weeks	4–28–43 745 Weeks	10–14–43 768 Weeks	4–26–44 794 Weeks	10–11–44 816 Weeks	4–25–45 843 Weeks	10–18–45 866 Weeks	4–4–44 773 Weeks	4–5–46 825 Weeks
3 by 3 by 5/16-in. Plain Steel Angles															
Electrogalvanized	0.52	Specimens removed, 1942[1]						Specimens removed, 1942[1]						<5R	<5R
Hot-dip galvanized	2.52	0R	TR–Y	0R	0R	0R	0R	TR–Y	TR–Y	TR–Y	TR–Y	5 Y	5 Y	0R	0R
Hot-dip aluminum	0.89	0R	0R	0R	0R	0D	0D	0D	0R	0D	0R	0D	0D	TR–Y	TR–Y
Cadmium-plated	0.09	Specimens removed, 1942[1]						Specimens removed, 1942[1]						99R	99R*
Electric Sherardized	1.38	100Y	100Y	100Y	0R–100Y	100Y	100Y							TR–R 90Y	TR–R 98Y
Lead-plated	...	60R*	90R*	90R*	80R*	80R*	<5R–85R*							60R*	90R*
3 by 3 by 5/16-in. Copper-Bearing Angles															
Electrogalvanized	0.37	Specimens removed, 1942[1]						Specimens removed, 1942[1]						10R	15R
Hot-dip galvanized	2.88	TR–Y	10Y	15Y	0R–10Y	10Y	3Y	TR–Y	TR–Y	TR–Y	TR–Y	5–Y	5Y	Missing	0R
Hot-dip aluminum	0.82	0R	0R	0R	0R	0D	0D	0D	0R	0D	0R	0D	0D	0R	0R
Cadmium-plated	0.24	Specimens removed, 1942[1]						Specimens removed, 1942[1]						45R	60R
Electric Sherardized	1.44	100Y	1R–99Y	100Y	5R	<5R–99Y	<5R–98Y							55Y	TR–R 98Y
Lead-plated	...	80R*	90R*	95R*	80R*	85R*	5R–85R*							60R*	90R*
2 by 1½ by 3/16-in. Plain Steel Angles															
Electrogalvanized	0.53	Specimens removed, 1942[1]						Specimens removed, 1942[1]						20R	50R (15R to 90R)
Electrogalvanized	0.50													50R	
Hot-dip galvanized	3.24	0R	0R	5–Y	0R	TR–Y	TR–Y–0R	<5R–5Y	5Y	5Y	TR–R–TR–Y	5–Y	10–Y	0R	0R
Hot-dip aluminum	0.75	0R	0R	0R	0R	0D	0D	0D	0R	0D	0R	0D	0R	3R*	15R*
Cadmium-plated	0.43	Specimens removed, 1942[1]						Specimens removed, 1942[1]						95R	95R
Electric Sherardized	1.21													30R–70Y	90R–10Y
Lead-plated	...	60R*	80R*	80R*	65R*	75R*	80R*	65R*	60R*	60R*	45R*	65R*	TR–R 70R*	50R*	50R*
2 by 1½ by 3/16-in. Copper-Bearing Angles															
Electrogalvanized	0.70	Specimens removed, 1942[1]						Specimens removed, 1942[1]						20R	50R (15R to 90R)
Electrogalvanized	0.76													20Y–25R	
Hot-dip galvanized	2.75	0R	0R	0R	0R	0R	TR–Y–0R	<5R–5Y	5Y	5–Y	TR–R–TR–Y	5–Y	10–Y	0R	0R
Hot-dip aluminum	0.50	0R	0R	0R	0R	0D	0D	0D	0R	0D	0R	0D	0R	TR–R*	15R*
Cadmium-plated	0.50	Specimens removed, 1942[1]						Specimens removed, 1942[1]						75R	90R
Electric Sherardized	1.15													100R	100R
Gas Sherardized	0.17													100R	100R
Lead-plated	...	75R*	80R*	80R*	80R*	80R*	TR–R–80R*							5R–60R*	5R–60R*

[1] These specimens were removed in the fall of 1942 when the racks were rebuilt.

2-IN. DIAMETER TUBULAR GOODS

Electrogalvanized	1.58	0R	0R	0R	0R	0R	0R	Specimens removed, 1942[1]						0R	0R
Hot-dip galvanized	2.51	0R	0R	0R	OR–TR–Y	TR–Y	TR–Y–0R	10–Y	10–Y	5–Y	5–Y	5–Y	5–Y	0R	0R
Hot-dip aluminum	4.73	0R	0R	0R	0R	0D	0D	TR–Y	TR–Y	0R	0R	0D	TR–R*	5R*	10R*
Cadmium-plated	0.23	Specimens removed, 1942[1]						Specimens removed, 1942[1]						60R	70R
Cadmium-plated	0.10													90R	95R
Electric Sherardized	0.29													100R	100R
Electric Sherardized	0.85													100Y	100Y
Gas Sherardized	0.66	100Y	100Y	100Y	1R–99Y	100Y	100Y							100Y	100Y
Lead-plated	...	50R*	90R*	95R*	90R*	75R*	<5R–95R*	70R*	80R*	TR–R 80R*	90R*	90R*	<5R–50R*	75R*	75R*

1-IN. DIAMETER TUBULAR GOODS

Electrogalvanized	1.62	0R	0R	0R	0R	0R	0R	Specimens removed, 1942[1]						0R	0R
Hot-dip galvanized	1.83	10–Y	50–Y	70–Y	0R–50Y	90–Y	50–Y							TR–Y	TR–Y
Hot-dip aluminum	1.83	0R	0R	0R	0R	0D	0D							<5R*	<5R*
Cadmium-plated	0.30	Specimens removed, 1942[1]												Specimens mixed	5R
Cadmium-plated	1.18														5R
Electric Sherardized	0.42														Specimens mixed
Electric Sherardized	0.92	5R–95Y	5R–95Y	7R–80Y	10R–90Y	5R–95Y	10R–90Y								
Gas Sherardized	0.72	10R–90Y	15R–85Y	30R	20R	25R–75Y	30R–70Y								
Calorized	...	TR–Y	100Y	100Y	0D	0D	Blk.								
Lead-plated	...	75R*	90R*	95R*	85R*	75R*	<5–R–85R*								60R*
Amaloy	...	60R*	65R*	65R*	85R*	75R*	TR–R–70R*								60R*

FORGED STEEL TOWER FITTINGS

Electrogalvanized															
Bolt	0.31	Specimens removed, 1942[1]						Specimens removed, 1942[1]						10R	
Nut	0.54														
Clamp	1.15														
Electrogalvanized															
Bolt	0.02													<5R	
Nut	0.01														
Clamp	0.46														
Hot-dip galvanized	2.67	0R	0R	5Y	0R–TR–Y	10Y	20Y							Missing	
Hot-dip galvanized	1.53	80Y	80Y	70Y	0R–80Y	40Y	20Y							20Y	Specimens Mixed[a]
Hot-dip aluminum	...	0R	0R	TR–Y	0R	0D	0D							75R*	
Cadmium-plated	0.40	Specimens removed, 1942.[1]												40R	
Electric Sherardized	0.50	100Y	TR–R 90Y	100Y	5R–95Y	10R–90Y	15R–85Y							100Y	
Gas Sherardized	...	Specimens removed, 1942[1]												10R–90Y	
Parkerized	...													100R	
Cadmium-plated	0.26													100R	
Lead-plated	...													100R	

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[1] These specimens were removed in the fall of 1942 when the racks were rebuilt.

[a] 40R* on hot dip aluminum; some electrogalvanized and hot dip zinc 0R.

TABLE IV.—REPORT ON INSPECTIONS OF METALLIC COATINGS ON HARDWARE, STRUCTURAL SHAPES, TUBULAR GOODS, ETC., EXPOSED TO ATMOSPHERE ON TEST RACKS AT FIVE LOCATIONS.—*Continued.*

Abbreviations and symbols used: 0, no rust; 5, 10, 25, 100, approximate percentages; R, rusting of base metal; Y, yellow or orange yellow rust; *, rust spots widely distributed; D, black or dark surface film (soot or dirt); a, in angles; <, less than; e, edges; TH, threads; TR, trace; SL, slight; N, near; S, stain; X, 100 per cent rust listed in previous reports.

Coating	Weight, oz. per sq. ft. of surface	Exposure at State College, Pa.						Exposure at Sandy Hook, N. J.						Exposure at Key West, Fla.	
		4-29-43 736 Weeks	10-13-43 758 Weeks	4-27-44 785 Weeks	10-10-44 808 Weeks	4-26-45 835 Weeks	10-17-45 858 Weeks	4-28-43 745 Weeks	10-14-43 768 Weeks	4-26-44 794 Weeks	10-11-44 816 Weeks	4-25-45 843 Weeks	10-18-45 866 Weeks	4-4-44 773 Weeks	4-5-46 825 Weeks
¾-in. Nipples and Ells															
Electrogalvanized MI															
{Nipple	1.64	0R	0R	0R	0R	TR-R	0R	Specimens removed, 1942[1]						15R	Specimens mixed[a]
{Ell	1.55														
Electrogalvanized CI															
{Nipple	2.02	10R	5R	<5R	5R	<5R	5R							15R	
{Ell	2.36														
Electrogalvanized MI															
{Nipple	2.43	10R	5R	<5R	5R	10R	15R							0R	
{Ell	2.90														
Electrogalvanized CI															
{Nipple	1.82	10R	10R	10R	20R	15R	10R							20R	
{Ell	0.67														
Hot-dip galvanized MI	1.82	85Y	100Y	90Y	100Y	90Y	50Y	Specimens removed, 1942[1]						TR-Y	
Hot-dip galvanized CI	1.44	85Y	60Y	70Y	95Y	80Y	50Y	5R-95Y	10R-60Y	15R-70Y	15R-85Y	15R-75Y	35R-50Y	TR-Y	
Hot-dip aluminum MI	3.20	R-TH	R-TH	R-TH	R-TH	R-TH	R-THD	R-TH	R-TH	R-TH	R-TH	R-TH	R-TH	45R	
Hot-dip aluminum CI	2.80	R-TH	0R	R-TH	0R	R-TH	R-TH	R-TH	R-TH	R-TH	R-TH	R-TH	R-TH	45R	
Cadmium-plated MI	...	Specimens removed, 1942[1]						Specimens removed, 1942[1]						100R	
Cadmium-plated CI	...													100R	
Cadmium-plated MI	...													100R	
Cadmium-plated CI	...													100R	
Electric Sherardized MI	0.63													100Y	
Electric Sherardized CI	0.47													100Y	
Gas Sherardized MI	0.78													70R-30Y	
Gas Sherardized CI	0.84													100R	
Parkerized MI	...													100R	
Parkerized CI	...													100R	
Electric Sherardized MI	0.75	100Y	100Y	100Y	0R	100Y	5R-95Y							100Y	
Electric Sherardized CI	0.82	Specimens removed, 1942[1]												60R-40Y	
Amaloy MI	...	95R*	95R*	100R*	100R*	90R*	100R*	95R*	95R*	100R*	90R*	90R*	TR-R-70R*	50R-50Y	
Amaloy CI	...	95R*	95R*	100R*	100R*	90R*	100R*	95R*	95R*	100R*	90R*	90R*	TR-R-70R*	50R-50Y	

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[1] These specimens were removed in the fall of 1942 when the racks were rebuilt.
[a] 40R* on hot-dip aluminum; some electrogalvanized and hot-dip zinc 0R.

Electrogalvanized MI														
Bolt	0.31	Specimens removed, 1942[1]						Specimens removed, 1942[1]						5R	
Nut	0.54													
Clamp	1.15													
Electrogalvanized CI														
Bolt	0.28												5Y	
Nut	0.56													
Clamp	...													
Electrogalvanized MI														
Bolt	0.02												10R-35Y	
Nut	0.01													
Clamp	0.46													
Electrogalvanized CI														
Bolt	0.04												15R-50Y	
Nut	0.04													Specimens mixed
Clamp	0.74													
Hot-dip galvanized MI	1.53												TR-R*	
Hot-dip galvanized CI	1.75												TR-R*	
Cadmium-plated MI	0.28												99R	
Cadmium-plated CI	0.28												100R	
Cadmium-plated MI	0.15												Specimens	
Cadmium-plated CI	0.17												0R mixed	
Hot-dip galvanized MI	2.00	10Y	5Y	5Y	TR-Y	10Y	10Y	15Y	60Y	50Y	60Y	0R	<5R-20Y	missing	
Hot-dip galvanized CI	2.38	10Y	5Y	5Y	TR-Y	10Y	10Y	TR-R	TR-R	TR-R	TR-R	TR-R	<5R-20Y		
Electric Sherardized MI	...	Specimens removed, 1942[1]						Specimens removed, 1942[1]						Specimens mixed	
Electric Sherardized CI	1.82													
Gas Sherardized MI	0.50													
Gas Sherardized CI	0.63													
Parkerized MI	...													Specimens mixed
Parkerized CI	...													
Hot-dip aluminum MI	3.16	0R	0R	0R	0R	0D	0D	0D	0R	0D	0R	0R	0R		
Hot-dip aluminum CI	2.51	0R	0R	0R	0R	0D	0D	0D	0R	0D	0R	0R	0R		
Amaloy MI	...	Specimens removed, 1942[1]						Specimens removed, 1942[1]							
Amaloy CI	...														

1½ BY 1½ BY ⅛-IN. PLAIN STEEL ANGLES

Electrogalvanized	0.47	Specimens removed, 1942[1]						Specimens removed, 1942[1]						20R	20R (0 to 30R)
Electrogalvanized	1.09												5R	
Hot-dip galvanized	3.16	0R	0R	0R	0R	0R	0R	TR-Y	0R	TR-Y	TR-Y	5Y	TR-R-5Y	0R	0R
Hot-dip aluminum	0.89	0R	0R	0R	0R	0D	0D	0D	TR-Y	0D	0R	0D	0D	TR-R*	1R
Cadmium-plated	0.12	Specimens removed, 1942[1]						Specimens removed, 1942[1]						60R	65R
Electric Sherardized	0.14													100Y	100Y—Near R
Gas Sherardized	...													100R	100R
Lead-plated	...	40R*	50R*	90R*	70R*	50R*	70R*	65R*	50R*	TR-R-60R*	70R*	TR-R-50R*	<5R-70R*	50R*	60R*

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[1] These specimens were removed in the fall of 1942 when the racks were rebuilt.

TABLE IV.—REPORT ON INSPECTIONS OF METALLIC COATINGS ON HARDWARE, STRUCTURAL SHAPES, TUBULAR GOODS, ETC., EXPOSED TO ATMOSPHERE ON TEST RACKS AT FIVE LOCATIONS.—*Continued.*

Abbreviations and symbols used: 0, no rust; 5, 10, 25, 100, approximate percentages; R, rusting of base metal; Y, yellow or orange yellow rust; *, rust spots widely distributed; D, black or dark surface film (soot or dirt); a, in angles; <, less than; e, edges; TH, threads; TR, trace; SL, slight; N, near; S, stain; X, 100 per cent rust listed in previous reports.

Coating	Weight, oz. per sq. ft. of surface	Exposure at State College, Pa.						Exposure at Sandy Hook, N. J.						Exposure at Key West, Fla.	
		4–29–43 736 Weeks	10–13–43 758 Weeks	4–27–44 785 Weeks	10–10–44 808 Weeks	4–26–45 835 Weeks	10–17–45 858 Weeks	4–28–43 745 Weeks	10–14–43 768 Weeks	4–26–44 794 Weeks	10–11–44 816 Weeks	4–25–45 843 Weeks	10–18–45 866 Weeks	4–4–44 773 Weeks	4–5–46 825 Weeks
1½ by 1½ by ⅛-in. Copper-Bearing Angles															
Electrogalvanized	0.76	Specimens removed, 1942[1]						Specimens removed, 1942[1]						10R	20R
Electrogalvanized	0.47													60R–20Y	75R
Hot-dip galvanized	3.20	0R	0R	0R	0R	TR–Y	TR–Y–0R	TR–Y	5Y	TR–Y	TR–Y	5Y	5Y	0R	0R
Hot-dip aluminum	1.11	0R	0R	0R	0R	0D	0D	0D	0R	0D	0R	0D	0D	TR–R*	5R*
Cadmium-plated	0.65	Specimens removed, 1942[1]						Specimens removed, 1942[1]						35R	50R
Electric Sherardized	...													20R–80Y	20R–80Y
Gas Sherardized	...													Missing	Missing
Lead-plated	...	70R*	85R*	90R*	85R*	75R*	TR–R–95R*	50R*	40R*	TR–R–40R*	35R*	40R*	TR–R–40R*	30R*	60R*
No. 14 Gage by 2½-in. Plain Steel Flats															
Electrogalvanized	0.45	Specimens removed, 1942[1]						Specimens removed, 1942[1]						20R	40R
Electrogalvanized	0.70													5Y	20R
Hot-dip galvanized	3.16							10Y	TR–R–10Y	TR–R–5Y	TR–R–5Y	TR–R–10Y	5R–10Y	0R	0R
Hot-dip aluminum	0.89							0D	0R	0D	0R	0R	0R	TR–R*	TR–R*
Cadmium-plated	...							Specimens removed, 1942[1]						40R	75R
Electric Sherardized	0.58													20R–80Y	40R60Y
Gas Sherardized	...													15R–85Y	20R80Y
Lead-plated	...							50R*	35R*	35R*	45R*	40R*	TR–R–35R*	60R*	60R*
No. 14 Gage by 2½ in. Copper-Bearing Steel Flats															
Electrogalvanized	0.29	Specimens removed, 1942[1]						Specimens removed, 1942[1]						35R	35R
Electrogalvanized	0.62													30R	80R
Hot-dip galvanized	3.20	0R	0R	TR–Y	0R	TR–Y	TR–Y–0R							0R	0R
Hot-dip aluminum	0.47	0R	0R	0R	0R	0D	0D							TR–R*	15R*
Cadmium-plated	...	Specimens removed, 1942[1]												95R	98R*
Electric Sherardized	...													25R–75Y	15R85Y
Gas Sherardized	...													25R–75Y	90R10Y
Lead-plated	...	40R*	50R*	50R*	50R*	50R*	45R*							60R*	60R*

[1] These specimens were removed in the fall of 1942 when the racks were rebuilt.

¾ by 6-in. Bolts				
Sherardized	1.10	Specimens removed, 1942[1]	Specimens removed, 1942[1]	100R
Sherardized	0.68			55R*
Hot-dip galvanized	1.61			Missing
Electrogalvanized	0.33			100R
Cadmium-plated	0.29			100R
Cadmium-plated	0.22			60R
⅝ by 2½-in. Bolts				
Sherardized	0.87	Specimens removed, 1942[1]	Specimens removed, 1942[1]	25R*
Sherardized	0.70			100R
Hot-dip galvanized	1.40			TR-R
Cadmium-plated	...			25R
Cadmium-plated	...			100R
Electrogalvanized	0.20			99R
½ by 2½-in. Bolts				
Sherardized	0.84	Specimens removed, 1942[1]	Specimens removed, 1942[1]	Specimens mixed
Sherardized	0.79			
Hot-dip galvanized	1.48			
Electrogalvanized	0.21			
Electrogalvanized	0.20			
Cadmium-plated	0.30			
⅜ by 1½-in. Bolts				
Sherardized	1.20	Specimens removed, 1942[1]	Specimens removed, 1942[1]	100R
Sherardized	0.89			20R*
Hot-dip galvanized	1.45			TR-R*
Electrogalvanized	0.18			100R
Cadmium-plated	0.14			85R
Cadmium-plated	0.15			100R
1-in. Roofing Nails				
Sherardized	0.90	Specimens removed, 1942[1]	Specimens removed, 1942[1]	80R 20Y
Sherardized	0.86			Missing
Cadmium-plated	...			Missing
Hot-dip galvanized	1.34			0R
Hot-dip galvanized	1.15			0R
Electrogalvanized	...			0R
Amaloy	...			70R*
No. 14 by 2½-in. Wood Screws				
Sherardized	0.94	Specimens removed, 1942[1]	Specimens removed, 1942[1]	Specimens mixed
Sherardized	0.86			
Cadmium-plated	0.07			
Hot-dip galvanized	1.26			
Cadmium-plated	0.04			
Hot-dip galvanized	...			
Parkerized	...			
Electrogalvanized	...			

¾ by 6-in. Bolts	
Sherardized	100R
Sherardized	Specimens mixed
⅝ by 2½-in. Bolts	
Sherardized	100Y
Sherardized	100R
Hot-dip galvanized	0R
Cadmium-plated	90R
Cadmium-plated	90R
Electrogalvanized	100R
½ by 2½-in. Bolts	
Sherardized	Specimens mixed[b]
⅜ by 1½-in. Bolts	
Sherardized	Specimens mixed[b]
1-in. Roofing Nails	
Sherardized	100R
Sherardized	Missing
Cadmium-plated	Missing
Hot-dip galvanized	0R
Hot-dip galvanized	0R
Electrogalvanized	0R
Amaloy	95R*
No. 14 by 2½-in. Wood Screws	
Sherardized	Specimens mixed[c]

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[1] These specimens were removed in the fall of 1942 when the racks were rebuilt.
[b] Hot-dip zinc, 0R.
[c] Hot-dip zinc, 0R; Sherardized, 100Y; cadmium-plated, 0R to 50R.

TABLE IV.—REPORT ON INSPECTIONS OF METALLIC COATINGS ON HARDWARE, STRUCTURAL SHAPES, TUBULAR GOODS, ETC., EXPOSED TO ATMOSPHERE ON TEST RACKS AT FIVE LOCATIONS.—*Continued.*

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KNOCKOUT BOXES															
Electrogalvanized	0.21	Specimens removed, 1942[1]						Specimens removed, 1942[1]						TR-R	TR-R
Electrogalvanized	0.16													20R	20R
Hot dip galvanized	1.56	10Y	10Y	5Y	5Y	10Y	5Y	70Y	80Y	75Y	90Y	90Y	<5R-90Y	0R	TR-Y
Cadmium-plated	0.12	Specimens removed, 1942[1]						Specimens removed, 1942[1]						100R	100R
Cadmium-plated	0.19													30R	50R
Electric Sherardized	0.28	100Y	100Y	100Y	100Y	100Y	100Y							100R	100R
Parkerized		Specimens removed, 1942[1]												10R-90Y	100R
Gas Sherardized	0.19													100Y	100Y
Electric Sherardized	0.28	100Y	100Y	100Y	100Y	100Y	100Y							100R	100R
Amaloy	...	80R*	90R*	100R*	90R*	85R*	<5R-95R*	95R*	90R*	100R*	97R*	95R*	TR-R-50R*	100R	98R*
Hot-dip aluminum	1.84	0D	0R	Damaged	Damaged	0D	Damaged	0D	0R	0D	0R	TR-Y-0R	0R	5R*	15R*
STEEL HINGES															
Cadmium-plated	0.29	Specimens removed, 1942[1]						Specimens removed, 1942[1]						80R	85R
Cadmium plated	0.32													50R	85R
Hot-dip galvanized	1.54													Missing	Missing
Electric Sherardized	0.40													100R	100R
Gas Sherardized	0.68													2R-98Y	2R-98Y
Parkerized														100R	100R
Electrogalvanized	0.19													10R-90Y	10R90Y
BULLDOG CLAMPS															
Electrogalvanized	3.41	Specimens removed, 1942[1]												0R	0R
Hot-dip galvanized	1.88							5R-90Y	5R-95Y	10R-90Y	20R-80Y	10R-90Y	25R-75Y	TR-Y	TR-Y
Cadmium-plated	0.22							Specimens removed, 1942[1]						100R	100R
Cadmium-plated	0.17													100R	100R
Gas Sherardized	0.46													Missing	Missing
Electric Sherardized	1.25													100R	100R
Electric Sherardized	0.58													100Y	100Y
Hot-dip aluminum	1.35	0D	TR-R	TR-R	<5R	<5R	<5R	0D	0R	0D	0R	TRY-0R	0R	35R*	40R*
Parkerized	...	Specimens removed, 1942[1]						Specimens removed, 1942[1]						100R	100R

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[1] These specimens were removed in the fall of 1942 when the racks were rebuilt.

Electrogalvanized	0.76	Specimens removed, 1942[1]						Specimens removed, 1942[1]						10R	30R
Electrogalvanized	0.45													0R Damaged	30R
Hot-dip galvanized	2.49	0R	0R	0R	0R–TRY	TR–Y	TRY–0R	TR–R–5Y	5R–10Y	TR–Y	Damage 10Y	10Y	5R–10Y	5R*	0R
Hot-dip aluminum	0.90	0R	0R	0R	0R	0D	0D	0D	0R	0D	0R	0D	0D	TR-R*	TR-R*
Cadmium-plated	0.23	Specimens removed, 1942[1]						Specimens removed, 1942[1]						40R	60R
Electric Sherardized														5R 95Y	30R70Y
Gas Sherardized	0.12													100R	100R
Lead-plated	...	80R*	80R*	95R*	90R*	80R*	85R*	35R*	30R*	25R*	40R*	TR-R-40R*	TR-R-40R*	60R*	60R*

¼ BY 3-IN. COPPER-BEARING FLATS

Electrogalvanized	0.84	Specimens removed, 1942[1]						Specimens removed, 1942[1]						TR-Y	<5R
Electrogalvanized	0.66	0R	TR-R	10Y	7R	10R	20R	0D	0R	0D	0R	0D	0D	1-Y	<5R
Hot-dip galvanized	2.67	0R	0R	0R	0R	0R	0R	5Y	10Y	5Y	TR-Y	10Y	TR-R-5Y	0R	0R
Hot-dip aluminum	0.66	2R	0R	0R	0R	0D	0D	0D	0R	0D	0R	0D	0D	TR-R*	2R*
Cadmium-plated	0.27	Specimens removed, 1942[1]						Specimens removed, 1942[1]						60R	80R
Electric Sherardized														10R-90Y	25R75Y
Gas Sherardized	0.18													100R	100R
Lead-plated	...	50R*	60R*	75R*	70R*	75R*	TR-R-70R*	40R*	40R*	35R*	40R*	TR-R-35R*	TR-R-40R*	60R*	60R*

No. 209 WINDOW FRAME SECTION

Electrogalvanized	0.67	Specimens removed, 1942[1]	Specimens removed, 1942[1]	35R	40R
Electrogalvanized	0.24			35R	45R
Hot-dip galvanized	3.10			0R	0R
Hot-dip aluminum	0.61			TR-R*	5R*
Cadmium-plated	0.24			80R	95R
Electric Sherardized	0.52			10R-85Y	15R85Y
Amaloy	...			60R*	60R*

No. 792 WINDOW FRAME SECTION

Electrogalvanized	0.54	Specimens removed, 1942[1]						Specimens removed, 1942[1]						20R	20R
Electrogalvanized	0.35													80R	95R
Hot-dip galvanized	2.60	0R	0R	0R	0R	0R	0R	10Y	30Y	15Y	TR-R-5Y	TR-R-10Y	TR-R-15Y	0R	0R
Hot-dip aluminum	1.39	0R	0R	0R	0R	0D	0D	Specimens removed, 1942[1]						TR-R*	TR-R*
Cadmium-plated	0.22	Specimens removed, 1942[1]												35R	50R
Cadmium-plated	0.17													80R	95R
Electric Sherardized	0.57													100Y	100Y
Gas Sherardized	0.53													40R-60Y	50R50Y
Parkerized	...													100R	100R

No. 161 WINDOW FRAME SECTION

Electrogalvanized	0.89	Specimens removed, 1942[1]	Specimens removed, 1942[1]	Missing	98R (3 Missing)
Electrogalvanized	0.81			Missing	35R
Hot-dip galvanized	2.97			0R	0R
Hot-dip aluminum	0.66			5R*	5R*
Cadmium-plated	0.22			Specimens mixed	90R
Cadmium-plated	0.15				90R (2 Missing)
Electric Sherardized	0.32				100Y (1 Missing)
Gas Sherardized	0.42				20R80Y
Parkerized	...				100R

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[1] These specimens were removed in the fall of 1942 when the racks were rebuilt.

of the electrogalvanized and Sherardized specimens had rusted completely and were not replaced. A few specimens carrying heavy coatings of these types remain on test. The aluminum-coated specimens are free from rust except at threaded areas where the threads had been recut due to roughness of the coating. The lead-coated specimens do not show much change in the percentage area of pinhole rusting characteristic of this coating. The percentage estimates vary, probably due more to the inspectors' judgment than actual changes in appearance. A few sets of the lead-coated specimens now show small areas (trace to 5 per cent) of ordinary deep-seated scale rust in addition to the pinhole rusting.

TABLE V.—CORROSION CONDITIONS OF ZINC-COATED UNFABRICATED WIRES AFTER APPROXIMATELY NINE YEARS' EXPOSURE.

R, rust; Y, yellowed; G, gray; M, metallic; MG and GY, intermediate states.

Coating Group, oz. per sq. ft.	Pittsburgh, Pa.	Sandy Hook, N. J.	Bridgeport, Conn.	State College, Pa.	Lafayette, Ind.	Ames, Iowa	Manhattan, Kans.	Ithaca, N. Y.	Santa Cruz, Calif.	College Station, Tex.	Davis, Calif.
0.20 to 0.30	100R	100R	100R	100R	100R	100R	Y	100R	GY	4R	G
0.25 to 0.35	100R	100R	100R	100R	100R	64R	4R	98R	GY	2R	G
0.35 to 0.45	100R	100R	100R	100R	100R	17R	GY	100R	GY	GY	G
0.45 to 0.55	100R	100R	100R	40R	87R	1R	GY	27R	GY	GY	G
0.50 to 0.60	100R	100R	100R	20R	46R	1R	MG	19R	MG	G	MG
0.60 to 0.70	100R	100R	100R	2R	24R	G	G	1R	G	G	GY
0.70 to 0.85	100R	100R	100R	GY	13R	G	MG	G	MG	G	MG
0.80 to 1.00	100R	87R	95R	GY	1R	G	MG	G	MG	G	MG
1.20 to 1.35	100R	2R	25R	G	G	G	MG	G	...	G	...
1.60 to 1.85	100R	1R	1R	G	G	G	MG	M	MG	G	MG
2.00 or more	99R	G	G	G	G	G	MG	M	MG	G	MG

TABLE VI.—NUMBER OF WIRE SPECIMENS REMOVED FOR TENSION TESTS.

Location	Number of Wires Removed in 1945					Wires Removed Prior to 1945	Total Removed to Date
	Bare Steel	Zinc-Coated	Lead-Coated	Copper-Coated	Chrome, Nickel Steel		
Pittsburgh, Pa	0	15	0	2	10	546	573
Sandy Hook, N. J.	0	52	0	0	0	207	259
Bridgeport, Conn.	3	56	0	0	0	169	228
State College, Pa.	0	0	0	0	0	88	88
All other locations	0	0	0	0	0	282	282
Total	3	123	0	2	10	1292	1430

Sandy Hook, N. J.:

As at State College, only zinc, aluminum, and lead-coated specimens remain on exposure. Several of the hot-dip zinc-coated specimens show red rust and most of them yellow rust. A few of the hot-dip aluminum-coated specimens showed a yellow pinhole type of rust, but not always at consecutive inspections. There may be some sealing and reopening of pores in the coating since

TABLE VII.—LOSS OF WEIGHTS OF ZINC-COATED WIRES EXPOSED AT STATE COLLEGE, PA. (8.29 YR.)
Tests on Unfabricated Wires Weighed and Measured Before Exposure—Losses of Weight in Ounces per Square Foot

Gage of Wire	Group A[a]		Group B[a]		Group C[a]		Average of Like Gages
	Wire Lot	Loss[b]	Wire Lot	Loss[b]	Wire Lot	Loss[b]	
No. 6	No. 148	0.507					0.507
No. 9			No. 247	0.526	No. 248	0.419	0.472
No. 11	No. 346	0.527	No. 345	0.549	No. 348	0.434	0.503
No. 14½	No. 542	0.563	No. 541	0.548	No. 540	0.410	0.540
	Average	0.532	Average	0.541	Average	0.421	0.498

[a] By reference to the published referee test results, the wires listed under Group A (or B, or C) will be found to be closely alike as regards base-metal composition and coating characteristics.

[b] Each weight-loss figure is the average of four specimens of each wire lot number.

there is present on these specimens a white bloom of corrosion products sometimes covered by a dark layer. The aluminum-coated specimens show rusting at recut threads. The lead-coated specimens continue to show the pinhole type of rust over a large percentage of the surface area and some additional sets now show the start of normal scale rusting.

Key West, Fla.:

All of the original specimens remain on exposure at this location. However, complete rusting had previously occurred on all Parkerized samples and some of the thinly coated electrogalvanized, Sherardized, and cadmium-plated specimens. At the April, 1946, inspection: (*1*) most of the hot-dip galvanized specimens were still free from rust, (*2*) nearly all hot-dip aluminum-coated specimens show pinhole rusting and a heavy white corrosion crust, and (*3*) the lead-coated specimens continue to show pinhole rusting characteristic of this type of coating with less tendency toward scale rusting than at the northern locations.

EXPOSURE TESTS OF WIRE AND WIRE PRODUCTS

Regular semiannual inspections of the specimens on exposure at Pittsburgh, Sandy Hook, Bridgeport, and State College were continued in 1945. The regular annual inspections at the university test plots were made by the resident university people. The findings from these visual inspections are summarized in Table V.

During 1945 there were removed 138 tension specimens, as shown in Table VI. The tension tests on the wires which were originally zinc-coated and are now rusting do not show any significant change in the rate of strength loss previously noted for such wires. The copper-covered wires, and the chrome-nickel wires removed from the Pittsburgh test plot (the most corrosive site) do not show any loss in tensile strength as yet. No lead-coated wires were removed for test in 1945.

In addition to the above removals, the last remaining set (36 wires) of weight-loss specimens was removed from State College. The results from these weight-loss specimens are shown in Table VII. The over-all average annual loss of coating in ounces per square foot of surface for this group at State College is 0.060. This concurs with the previously published figure for this site.

This report has been submitted to letter ballot of Subcommittee VIII consisting of 67 members; 49 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the subcommittee,

C. E. REINHARD,
Chairman.

REPORT OF COMMITTEE A-6[*]

ON

MAGNETIC PROPERTIES

Due to traveling conditions, only one meeting of Committee A-6 on Magnetic Properties has been held since the 1944 Annual Meeting; however, it has been found possible to carry on much of the work by correspondence. No report was submitted last year.

Since the previous report in 1944, the following additions have been made to the committee personnel: Cecil J. Bier of the Biad Powder Metallurgy Co.; Patrick E. Cavanagh of the Allen B. DuMont Labs.; Gilbert Feth of the Follansbee Steel Corp.; C. C. Horstman of the Westinghouse Electric Corp.; and a representative of the Navy Department. J. S. Murray and S. L. Burgwin have resigned. The loss of A. V. de Forest by death, is deeply regretted by the committee.

Since the previous report, the 15-kilogauss test for core loss has been generally adopted by industry as a standard for acceptance tests of electrical sheet. The 10-kilogauss test is still optional, except for oriented grain material for which the higher induction test only is used.

Work on the development of a standard form for preparing specifications for electrical sheet has continued. A much improved form is now before the committee for consideration.

In view of the fact that some of the tests specified in A 34 involve properties other than magnetic, the Executive Committee of the Society has been asked to approve a change in the title of Committee A-6 to read: "Committee A-6 on Magnetic Materials." Also a change in the scope of the Committee was suggested, as follows:

> *Scope.*—The promotion of knowledge of the properties of magnetic materials and the formulation of standards relating thereto.

The present officers and members of the Advisory Committee have been reelected for the ensuing term of two years.

I. NEW TENTATIVE

The committee recommends for publication as tentative new Methods of Test for Core Loss at Frequencies Higher than Power Frequencies and Ductility of Magnetic Materials,[1] to be eventually incorporated in Standard Methods A 34. The test procedures for core losses at frequencies from 200 to 2000 cycles were prepared following a series of round-robin tests which showed remarkably good agreement. The test procedures for ductility cover the Olsen or Erichsen cup test and a bend test for use at the option of the purchaser or seller.

II. ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that the Tentative Definitions of Terms, with Symbols, Relating to Magnetic Testing (A 127 - 44 T)[2] be approved for reference to letter ballot of the Society for adoption as standard with the following revisions:

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[*] Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] This method was accepted as tentative by the Society and appears in the 1946 Book of A.S.T.M. Standards, Part I-A.

[2] 1944 Book of A.S.T.M. Standards, Part I.

Coercive Force, Intrinsic, H_{ci}.—Change to read as follows: "The magnetizing force required to bring to zero intrinsic induction of a magnetic material which is in a symmetrically cyclically magnetized condition."

Induction, Magnetic (Magnetic Flux Density), B.—In the beginning of the third paragraph, change "Under a-c. conditions" to read "Under sinusoidal conditions."

Add the following new definitions:

Coercive Force, Relaxation.—The reversed magnetizing force of such a value that when reduced to zero, the induction becomes zero.

Exciting Current, a-c.—The rms. value of the total current supplied to a coil, linked with a magnetic core.

Magnetizing Current, a-c.—The rms. value of the quadrature component (with respect to the induced voltage) of the exciting current supplied to a coil, linked with a magnetic core.

Magnetic Particle Method.—In order to conform to the definition in Section 2 of the Tentative Method of Magnetic Particle Testing and Inspection of Heavy Forgings (A 275 - 44 T), change to read as follows: "A method for detecting defects on or near the surface in suitably magnetized materials, which employs finely divided magnetic particles that tend to congregate in regions of magnetic nonuniformity associated with the defects."

III. REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption a revision of the Standard Methods of Test for Magnetic Properties of Iron and Steel (A 34 - 44), as appended hereto,[3] and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that this recommendation may be referred to letter ballot of the Society.

IV. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD

The committee recommends that the tentative revision[4] of the Standard Methods of Test for Magnetic Properties of Iron and Steel (A 34 - 44), issued in 1944, be approved for reference to letter ballot of the Society for adoption as standard. These are minor changes, dealing primarily with the size of lots and provisions for additional tests in case of failure of the regular samples.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE			
Test for Core Loss at Frequencies Higher than Power Frequencies and Ductility of Magnetic Materials (A 34 - —T)	24	0	0
II. ADOPTION OF TENTATIVE AS STANDARD			
Def. of Terms, with Symbols, Relating to Magnetic Testing (A 127 - 44 T), as revised	24	0	0
III. REVISION OF STANDARD, IMMEDIATE ADOPTION			
Tests for Magnetic Properties of Iron and Steel (A 34 - 44)	24	0	0
IV. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD			
Methods of Test for Magnetic Properties of Iron and Steel (A 34 - 44)	24	0	0

The recommendations appearing in this report have been submitted to letter ballot of the committee, which consists of 26 members; 24 members returned their ballots, with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Definitions (P. H. Dike, chairman).—No meetings of this subcommittee have been held since the previous report in 1944. However, a few changes in existing definitions, and a few new ones have been developed by correspondence, as indicated earlier in this report. This committee has also acted as an Editorial Committee with reference to the Standard Methods A 34 which have been completely revised.

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[3] This revised method was accepted by the Society and appears in the 1946 Book of A.S.T.M. Standards, Part I-A.

[4] 1944 Book of A.S.T.M. Standards, Part I, p. 2003.

Subcommittee III on Direct Current Test Methods (B. M. Smith, chairman).—This committee has considered a new method of testing feebly magnetic materials, as suggested by Subcommittee VIII of Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys. No decision has been made concerning its inclusion in Standard Methods A 34. The committee expects soon to make recommendations with reference to the use of the 25-cm. frame for d-c. testing. It is hoped that this method will supplant the use of permeameters for laminated materials, excepting for test at high-magnetizing forces.

Subcommittee IV on Alternating Current Test Methods (J. P. Barton, chairman).—This subcommittee has been very active, carrying on its work mostly by correspondence. As a result, new tentatives dealing with ductility tests, methods of measuring core losses at higher than power frequencies, and minor changes in standard test procedures have been prepared. A method for testing the resistivity of electrical sheet has also been developed and is included in Standard Methods A 34. If it seems desirable, this will later be correlated with methods in use which were developed by other committees of the Society. It is desirable to have a suitable methods since core losses are to a considerable extent a function of the electrical resistance of magnetic materials.

This report has been submitted to letter ballot of the committee, which consists of 26 members; 24 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

THOMAS SPOONER,
Chairman.

R. L. SANFORD,
Secretary.

REPORT OF COMMITTEE A-10*

ON

IRON-CHROMIUM, IRON-CHROMIUM-NICKEL AND RELATED ALLOYS

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys held a meeting in Pittsburgh, Pa., on February 26, 1946.

The committee consists of 83 members, of whom 37 are classified as producers, 34 as consumers, and 12 as general interest members.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, Jerome Strauss.
Vice-Chairman, H. L. Maxwell.
Secretary, H. D. Newell.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revision in the Recommended Practice for Conducting Plant Corrosion Tests (A 224 – 41)[1] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting that these modifications may be referred to letter ballot of the Society:

Section 1.—Letter this section as (*a*) and add the following as a new paragraph (*b*):

> (*b*) Tests of the sort considered here are probably the best means available for approximating the behavior of metals in service short of actually constructing and operating a piece of equipment. With proper consideration in setting up the test and interpreting the results, useful information can be obtained. Certain precautions must be recognized for best results. These include:
>
> (*1*) Specimens immersed in a hot liquid may not corrode at the same rate or in the same manner as in equipment where the metal acts as a heat transfer surface in heating or cooling the product. This restriction also applies to specimens exposed at ambient temperature in a gas stream from which water or other corrodants might condense on cooled surfaces.
>
> (*2*) Effects due to high velocity, abrasive ingredients, etc., in the product which may be emphasized in pipe elbows, pumps, etc., may not easily be reproduced.
>
> (*3*) Corrosion products from a particular metal may have undesirable effects on the product even though the total corrosion is small. This possibility is frequently recognized in advance. The extent of possible contamination can be estimated from the weight loss of the specimen with proper application of the expected relationships among the area of corroding surface, the mass of the product handled, and the duration of contact of a unit of mass of the product with the corroding surface.
>
> (*4*) Corrosion products from the metal of the plant equipment used in the test may influence the corrosion of one or more of the test metals; for example, if corrosion-resistant steel specimens were to be exposed in copper equipment where the copper is corroding.
>
> (*5*) The possibility that the corrosion products from one material may influence the corrosion of adjacent specimens of other materials should be considered. A random arrangement of multiple specimens may provide an indication of such effects.

Section 2 (*f*).—In line 2, after "galvanic effects" add the words "including galvanic protection."

Section 2 (*g*).—Delete Paragraph (*g*) relettering the subsequent paragraphs accordingly.

Section 3 (*a*).—At the end of item (*1*), add the words parenthetically "(see Item (*3*))"; also, add the following to Item (*3*): "Excessively thin specimens, which might affect the test results by edge or flexure effects, should be avoided. A minimum thickness of $\frac{1}{70}$ of the longest dimension is suggested. Perforation or excessive corrosion of thin specimens may not be cause for rejection. Retesting with thicker pieces may be desirable."

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] 1944 Book of A.S.T.M. Standards, Part I.

Add new Items (*6*) and (*7*) to read as follows:

> (*6*) When the choice of material has been narrowed down in preliminary tests, further tests should involve consideration of the effects of such factors of construction and assembly as welding, soldering, cold working, etc.
>
> (*7*) Tension or other specimens may be used to secure special information as on the existence of subsurface corrosion. Special methods such as described by Searle and LaQue[a] may be used for the installation of such specimens in tubes and other containers.

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[a] H. E. Searle and F. L. LaQue, "Corrosion Testing Methods," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 249 (1935).

Section 4 (*c*).—Change to read as follows: "(*c*) After the specimens are cut to size, they should be freed from water breaks by suitable cleaning. It is preferable to expose specimens with the surface representative of that likely to be used. The cleaning method should take this into consideration."

Section 5.—Delete the present note and add the following as a new note: "NOTE.—If an accuracy of plus or minus 10 per cent is adequate for the purpose of the test, duplicate specimens will suffice. If better accuracy is needed, the number of specimens of each material should be increased to 4 or 5."

Section 11 (*c*).—Change the third sentence to read as follows: "Sometimes a pit type of corrosion is initiated, but is self-healing and stops."

Section 11 (*f*).—In line 5, after "effects" add the words "including galvanic protection."

Section 13 (*c*).—Change to read as follows:

> (*c*) If there is any reason to believe that stress corrosion may be a consideration, supplementary tests may be made. These tests may at times be made in the plant equipment. The choice of specimen, specimen support, and the method of applying stress should be given careful consideration. In some instances, the technique described by Hoyt and Scheil[a] may be employed.

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[a] S. L. Hoyt and M. A. Scheil, "Stress-Corrosion Cracking in Austenitic Stainless Steels," *Transactions*, Am. Soc. Metals, Vol. 27, pp. 191–217 (1939).

Appendix, Section A1.—In Paragraph 2, after the second sentence, add the following: "Other suitable non-ferrous metals may also be used."

This recommendation has been submitted to letter ballot of the committee, which consists of 83 members; 66 members returned their ballots, of whom 61 voted affirmatively, 0 negatively, and 5 members marked their ballots "not voting."

EMERGENCY ALTERNATE PROVISIONS

The committee recommends the withdrawal of the following emergency alternate provisions:

EA – A 167a	Standard Specifications for Corrosion-Resisting Chromium-Nickel Steel Plate, Sheet, and Strip (A 167 – 44),
EA – A 177	Standard Specifications for High-Strength Corrosion-Resisting Chromium-Nickel Steel Sheet and Strip (A 177 – 44),
EA – A 268	Tentative Specifications for Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 – 44 T),
EA – A 269	Tentative Specifications for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 – 44 T),
EA – A 270	Tentative Specifications for Seamless and Welded Austenitic Stainless Steel Tubing for the Dairy and Food Industry (A 270 – 44 T), and
EA – A 271	Tentative Specifications for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 44 T).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Classification of

Data (E. F. Cone, chairman; Russell Franks, secretary).—The work of this subcommittee, which consists of compiling data on the cast alloys, has progressed to a point where the final compilation is ready for review by Committee A-10 and by the Alloy Castings Institute, following which it will be submitted to the Society for publication.

Subcommittee IV on Methods of Corrosion Testing (F. L. LaQue, chairman).—The principal activities of Subcommittee IV have been as follows:

(*1*) Working out details of a program of atmospheric exposure tests of corrosion-resistant steels, the final program having recently been submitted to the committee,

(*2*) Preparation for publication of a summary of information on the practice of Passivation of Corrosion-Resistant Steels by treatment with nitric acid, which appears in the Appendix.

(*3*) Preparation of a revision of Recommended Practice A 224 which is covered earlier, in this report.

(*4*) Organization of a symposium on Atmospheric Weathering of Corrosion-Resistant Steels[2] which includes seven papers on this subject which will be presented at the Annual Meeting of the Society this year. The principal continuing activity is the proposed program of atmospheric corrosion tests of corrosion-resistant steels which will be put into effect after the final program has been approved by the membership.

Subcommittee V on Mechanical Testing (T. F. Olt, chairman).—This subcommittee is still giving consideration to the techniques of mechanical testing of the corrosion-resistant steels with special reference to the effect of specimen shape and size and determination of yield strength properties.

Subcommittee VI on Metallography (Russell Franks, chairman).—This subcommittee is engaged in working out a new program relative to the etching and identification of sigma phase or other constituents in high-chromium and high-chromium - nickel steels. The formal program of the subcommittee's new activities will be presented at the June meeting.

Subcommittee VIII on Specifications for Bars, Forgings, and Wire (J. K. Findley, chairman).—This subcommittee has in preparation, specifications for 18 per cent chromium, 8 per cent nickel spring wire which specifications will be based on agreement between producers and consumers as to requirements for this product. Some minor changes may be made in the bar specifications with respect to the titanium and columbium content of the stabilized grades, and yield strength of type 410 material will be altered following review by the subcommittee. Future work will consist of the preparation of specifications for billets for forging.

Subcommittee IX on Specifications for Flat Products (H. A. Grove, chairman).—This subcommittee is recommending that Emergency Alternate Provisions of Specifications A 177 and A 167 be removed. Some changes in chemistry for grades appearing in Specifications A 167 are being considered.

The subcommittee recommends the following as an editorial change in the Standard Specifications for Corrosion-Resisting Chromium and Chromium-Nickel Steel Plate, Sheet, and Strip for Fusion-Welded Unfired Pressure Vessels (A 240 - 44):

Section 17.—Change the requirements for No. 1 Finish from their present form: namely,

> (*a*) Hot rolled, annealed, and pickled, or (*b*) Hot rolled, annealed, grit or sand blasted, and passivated.

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[2] See pp. 593 to 677.

to read as follows:

> (*a*) Hot rolled, annealed, and chemically descaled, or (*b*) Hot rolled, annealed, grit or sand blasted, and chemically cleaned.

Subcommittee X on Specifications for Castings (J. J. Kanter, chairman).—Subcommittee X is undertaking the revision of specifications for castings under its jurisdiction. The plan of revision consists of substituting two new designations embracing all of the apparently used grades and these designations will replace the current ones in the former casting specifications prepared for Committee A-10. One of these specifications will cover corrosion-resistant iron-chromium and iron-chromium-nickel alloy castings for general application and the second will cover heat-resisting iron-chromium and iron-chromium-nickel alloy castings for general application. The details concerning grades to be contained in the newly proposed specifications are in the hands of a Special Task Committee and will be available in the near future for ballot in the committee.

Subcommittee XI on Specifications for Tubular Products (J. J. B. Rutherford, chairman).—This subcommittee, in cooperation with members from Committee A-1 on Steel, is considering revision of the analysis of grades contained in Specifications A 268 and A 269. It has recommended the removal of the emergency alternate provisions for Specifications A 268, A 269, A 270, and A 271. Separately, the subcommittee is cooperating with Subcommittee XXII of Committee A-1 which has before it for consideration specifications for corrosion-resistant steel pipe, which will effect removal of these grades from the present Tentative Specifications for Seamless Alloy-Steel Pipe for High-Temperature Service (A 158 - 44 T). The subcommittee is maintaining contact with the Boiler Code Committee of The American Society of Mechanical Engineers with the ultimate object of providing specifications for austenitic corrosion-resistant steel tubing which will be suitable for A.S.M.E. Code use.

This report has been submitted to letter ballot of the committee, which consists of 83 members; 66 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

JEROME STRAUSS,
Chairman.

H. D. NEWELL,
Secretary.

EDITORIAL NOTE

The emergency alternate provisions listed in the report were discontinued by action of the Standards Committee on September 9, 1946.

Subsequent to the Annual Meeting, Committee A-10 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specifications for:

Corrosion Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (A 296 - 46 T), and

Heat Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (A 297 - 46 T).

These new tentatives were accepted by the Standards Committee on December 31, 1946, and appear in the 1946 Book of A.S.T.M. Standards, Part I-A bearing the designations indicated above.

APPENDIX I

REPORT ON PASSIVATION OF CORROSION-RESISTANT STEELS

Prepared by F. L. LaQue[1]

In June, 1944, Lt. Col. Greenall of the Frankford Arsenal asked the chairman of Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys to present before the members of the committee a problem dealing with passivation of corrosion-resistant steel and the appropriate procedures that ought to apply to such treatment for preventing corrosion and dimensional changes that are likely to be produced by such treatments. In further explanation of the problem he stated:

"This arsenal has received some conflicting views and claims that created some confusion as to whether passivating treatments are effective and whether such treatments are necessary. On this point the arsenal would like to have the committee's opinion in order to clarify the situation.

"The corrosion-resistant steels that this arsenal is particularly interested in and which it uses for fire-control instruments are of the martensitic, austenitic, and ferritic types. In brief, this arsenal would like to place before the committee the questions concerning the effectiveness and recommended procedures in passivating 18 per cent chromium, 8 per cent nickel type steels with or without stabilizers, 12 to 14 per cent chromium steel quenched and tempered, and heat-treatable straight chromium corrosion-resistant steel of the cutlery grade."

In order to elicit information on the points raised by Lt. Col. Greenall, the arsenal's request was circulated to members of Committee A-10 and answers were requested to certain questions which are summarized as follows:

Question 1:

What is likely to be accomplished by "the nitric acid treatment" of corrosion-resistant steels, loosely referred to as "passivation"?

Consensus:

Replies to this question emphasized that the prime function of the nitric acid "passivation" treatment was to clean the corrosion-resistant steel chemically and without harm. Such removal of free iron picked up during processing and fabrication was given special mention. Much less importance was attached to the possible reinforcement of the oxide films believed to be associated with passivity. It was pointed out that a properly cleaned corrosion-resistant steel surface rapidly would become passive in air and in many other environments and passivation is not necessary to make these alloys corrosion-resistant.

Question 2:

When is passivation, or acid cleaning, desirable?

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[1] Development and Research Division, The International Nickel Co., Inc., New York, N. Y.

Consensus:

(*a*) Whenever objectionable foreign material is known to be present, or suspected of being present.

(*b*) When it is desired for specific conditions to be certain that the surface of the corrosion-resistant steel will be in its most uniform and corrosion-resistant condition. This is likely to be a most important consideration when corrosive conditions are border-line in their attack.

(*c*) Before conducting corrosion tests.

(*d*) After mechanical cleaning or surface treatment, as with steel wool, wire brushes, grit blasting, and abrasives contaminated with iron. A possible exception is the case of material that has been buffed or polished, with abrasives in grease, followed by removal of excess grease and abrasive with powdered calcium carbonate or similar material.

Question 3:

What conditions as to nitric acid concentration, time, and temperature may have undesirable results with specific compositions and conditions of corrosion-resistant steel, as by:

(*a*) Corroding the corrosion-resistant steel so as to impair its finish, or removing sufficient metal to make it fail to meet tolerance specifications where these may be as close as 0.0001 in.?

(*b*) Failing to remove surface contamination by free iron—as by some passivating effect on such free iron?

Consensus:

(*a*) The free machining and high-carbon alloys, and particularly those of the straight chromium type, may be etched or dulled by hot weak solutions of nitric acid, such as under 5 per cent by volume boiling, 20 to 40 per cent by volume at 140 to 160 F., 15 per cent by weight at 135 F. It has been observed also that surfaces above the liquid level in the passivating bath are particularly likely to be etched under the indicated conditions. Low-chromium steels would also be attacked by boiling 65 per cent nitric acid.

(*b*) Concentrated solutions of nitric acid, for example 65 per cent by weight at atmospheric temperature, may passivate free iron and prevent its desired removal.

Question 4:

What are suitable conditions as to acid concentration (per cent by weight or volume to be specified), temperature, and time for the "passivation" or acid cleaning of the different grades and heat treatments of the corrosion-resistant steels mentioned by Lt. Col. Greenall?

Consensus:

The purposes of passivation may be accomplished under a great variety of conditions as to acid concentration, temperature, and time. Consequently, there is considerable latitude with respect to the treatments that may be used, especially in the case of the austenitic alloys. With the straight chromium steels, and especially the free machining, the 12 to 14 per cent chromium, and high-carbon types, there is some danger of superficial etching by hot, weak solutions. Therefore, it would appear to be desirable to place closer restrictions on the conditions of treatment of these latter steels.

Evidently it would be possible to cover the situation adequately as follows:

1. Passivate all the austenitic alloys and the straight chromium grades other than free machining compositions by treatment (complete immersion) in nitric acid solutions containing from 20 to 40 per cent by volume of concentrated nitric acid at a temperature from 130 to 160 F. for from 30 to 60 min., followed by rinsing in clean, hot water.

2. Passivate the free-machining straight chromium steels and those containing 12 to 14 per cent chromium by treatment in nitric acid solutions containing not less than 15 per cent by volume, and preferably 20 to 40 per cent by volume, nitric acid at a temperature not to exceed 150 F., for example 110 to 130 F. for 30 to 60 min.

3. In cases where steels show superficial etching as a result of the treatment by the nitric acid solutions suggested, an alternative treatment would be to use 20 per cent by volume nitric acid containing 2 per cent by weight sodium dichromate at 110 to 130 F. for 30 min. followed by immersion in 5 per cent by weight sodium dichromate solution at 140 to 160 F. for 1 hr.

4. In general, the desired results could be obtained as follows:

Acid concentration	20 to 40 per cent by volume of concentrated nitric acid for all compositions
Temperature	130 to 160 F. for the austenitic and straight chromium steels containing 17 per cent or more chromium 110 to 140 F. for the free-machining straight chromium steels and all grades containing 12 to 14 per cent chromium
Time	30 to 60 min. for all concentrations—the time might possibly be reduced to facilitate continuous operations

Material should be immersed completely.

In particular cases, the use of dichromate additions as covered by item 3 may be desirable.

Question 5:

What conditions should be maintained as to acid concentration, temperature and time which should be avoided for particular grades of corrosion-resistant steel where no surface attack during the treatment may be tolerated?

See Answers to Questions 3 and 4.

Question 6:

Are there any alternate methods for ridding corrosion-resistant steel surfaces of free iron—as by the use of some such detergent as naphtha soap?

Consensus:

Electropolishing accomplishes the purposes of passivation and need not be supplemented by any other passivation treatment. Electropolishing removes a significant amount of metal from the surface and therefore may not be a satisfactory substitute for other methods of passivation of finished parts that must be held to close tolerances on dimensions.

It has been found also that the nitric-hydrofluoric acid treatment (for example 8 to 10 per cent HNO_3 plus 1 to 2 per cent HF, followed immediately by thorough washing with water, preferably under pressure, or accompanied by mechanical scrubbing) commonly used as the final acid treatment on corrosion-resistant steels prior to shipment from the mills is sufficient and that nothing further is gained by an immediate additional treatment in a straight nitric acid solution.

The nitric-hydrofluoric acid solution may be operated at 140 to 160 F. on the chromium-nickel alloys, but should be used at room temperature for the chromium steels. Treatment time in either case should not be more than 5 to 7 min.

This solution is not recommended for polished or buffed finishes, nor for straight chromium steels containing less than approximately 17 per cent chromium with 0.15 per cent maximum carbon.

Question 7:

Do you think it would be desirable instead of specifying particular ways to "passivate" or acid clean corrosion-resistant steels to require only that surfaces be free from iron as tested by some reagent such as aceto acetanilide?

Consensus:

This was not considered to be a desirable alternative in view of the possible presence of undesirable contamination other than free iron.

Question 8:

Do you think that whether or not "passivation" is required as a finishing operation on corrosion-resistant steel parts it should be permitted on specimens to be subjected to acceptance tests, such as salt-spray tests?

Consensus:

Passivation of specimens to be subjected to acceptance tests probably should be required, or, at least permitted, except in such instances as the test may be used to detect the presence of free iron or other contamination that would ordinarily be removed by the "passivation" treatment. With particular regard to salt spray tests, it is pointed out that they are of doubtful significance and value as a means of measuring the quality of corrosion-resistant steel or its suitability for any particular purpose.

APPENDIX II

REPORT ON THE INSPECTION OF THE CORROSION-RESISTING STEEL DECK HOUSES ON THE U. S. NAVY DESTROYERS FARRAGUT AND ALWYN

A special subcommittee inspected the corrosion-resisting steel deck houses on two Navy destroyers, the Farragut No. 348 and the Alwyn No. 355, which have been in service since 1934 and 1935. The group making the inspection was as follows:

G. A. Pleam, Bureau of Ships, Navy Department
R. E. Wiley, Bureau of Ships, Navy Department
V. N. Krivobok, International Nickel Co.
G. A. Sands, Electro Metallurgical Co.
R. B. Gunia, Carnegie-Illinois Steel Corp.
M. D. V. Millard, American Steel and Wire Co.
P. G. Nelson, Edw. G. Budd Mfg. Co.
H. A. Grove, Republic Steel Corp., chairman.

Both of the above destroyers were fitted with corrosion-resisting steel deck houses made by the spot-welded process and fabricated by the Edward G. Budd Manufacturing Co. Some of the surrounding deck was also of corrosion-resisting steel. These destroyers have been in continuous service for approximately 10 to 11 years and served between the Aleutians and Australia during the greater part of the war. They were returned to the Brooklyn Navy Yard about the middle of October, 1945. The greater portion of these deck houses was constructed of corrosion-resisting steel but some sections were made both of carbon steel and aluminum.

An inspection of the corrosion-resisting steel in both the deck houses and the deck showed it to be in very good condition with no signs of corrosion. The deck houses as well as the deck had been painted but in a good many places this paint had broken away and there was every chance for sea water to condense under this paint. However, on removing additional paint no signs of corrosion or pitting were found.

The corrosion-resisting steel of these two deck houses was chiefly of the high tensile strength variety and was of spot-welded construction. None of these welds showed corrosive attack either of the pitting variety or stress corrosion. It was the understanding of the special subcommittee that no effort had been made to clean or passivate these welds after spot welding. All welds appeared to be in good sound condition.

On the Alwyn there was a large amount of corrosion-resisting steel used in the galley, and in lavatories in the crew's quarters. The corrosion-resisting steel in the galleys was in very good condition showing no rust or corrosion, while that in the lavatories had superficial rusting in some places but no pitting. All in all, the corrosion-resisting steel stood up very well and gave a good account of itself.

Respectfully submitted on behalf of the special subcommittee,

H. A. GROVE,
Chairman.

REPORT OF JOINT RESEARCH COMMITTEE

ON

EFFECT OF TEMPERATURE ON THE PROPERTIES OF METALS

PROGRESS REPORT TO THE SPONSOR SOCIETIES

During the past year the Joint Research Committee on Effect of Temperature on the Properties of Metals under the sponsorship of The American Society of Mechanical Engineers and the American Society for Testing Materials, held one meeting on November 28, 1945, in New York, N.Y., during the A.S.M.E. Annual Meeting. A second meeting was held on June 27, 1946, at Buffalo, N. Y., prior to the presentation of this report at the Annual Meeting of the A.S.T.M.

In addition, the committee sponsored a Session on Graphitization of Steel Piping held during the A.S.M.E Annual Meeting in New York, on November 28, 1945, comprising the following reports and papers:

"Summary Report on the Joint E.E.I.-A.E.I.C. Investigation of Graphitization of Piping," by S. L. Hoyt, R. D. Williams, and A. M. Hall.

"Progress Report for Project No. 29 on Susceptibility of Casting Steels to Graphitization," by J. J. Kanter.

"Comparative Graphitization of Some Low-Carbon Steels with and without Molybdenum and Chromium," by G. V. Smith, S. H. Brambir, and W. G. Benz.

"Further Observations of Graphitization in Aluminum-Killed Carbon-Molybdenum Steel Steam Piping," by R. W. Emerson, and Matthew Morrow.

"Graphitization in Some Cast Steels," by A. J. Smith, John B. Urban, and J. W. Bolton.

"Influence of Heat Treatment upon the Susceptibility and Graphitization of High Aluminum-Deoxidized Carbon-Molybdenum Steel," by F. Eberle.

The six papers and the discussion have been published by the A.S.M.E. in a special compilation, dated August, 1946, under the title "Graphitization of Steel Piping."

During the 1946 Annual Meeting of the A.S.T M. the committee sponsored two technical sessions, both on June 27, 1946. The first session was devoted to materials for gas turbines, and marks the public release of much of the confidential data developed by Government sponsored research of the war period. Five papers were presented, as follows:[1]

"Heat-Resisting Metals for Gas Turbine Parts," by Howard C. Cross and Ward F. Simmons.

"High-Temperature Alloys Developed for Aircraft Turbosuperchargers and Gas Turbines," by J. W. Freeman, E. E. Reynolds, and A. E. White.

"Chromium-Base Alloys," by Robert M. Parke and Frederick P. Bens.

"Metallurgy of High-Temperature Alloys used on Current Gas Turbine Designs," by F. S. Badger, Jr., and W. O. Sweeney, Jr.

"Alloys and Ceramic Materials for High-Temperature Service," by Howard C. Cross.

At the second session, five papers on various subjects were presented, as follows:

"The Effect of Carbide Spheroidization Upon the Rupture Strength and Elongation of Carbon-Molybdenum Steel,"[2] by S. H. Weaver.

"Compressive Properties of Aluminum Alloy Sheet at Elevated Temperatures,"[3] by Alan E. Flanigan, Leslie F. Tedsen, and John E. Dorn.

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[1] These papers are published in the Symposium on Materials for Gas Turbines, Am. Soc. Testing Mats. (Symposium issued as separate publication, November, 1946).

[2] See p. 856.

[3] See p. 951.

"Tensile and Creep Strengths of Some Magnesium-Base Alloys at Elevated Temperature,"[4] by A. A. Moore and John C. McDonald.

"Resistance of Iron-Nickel-Chromium Alloys to Corrosion in Air at 1600 to 2200 F.,"[5] by Anton deS. Brasunas, James T. Gow, and Oscar E. Harder.

"Influence of Strain Rate and Temperature on the Mechanical Properties of Monel Metal and Copper,"[6] by D. J. McAdam, Jr., G. W. Geil, and D. H. Woodard.

Finances:

There appears in Table I a summary of the administration of committee funds during the period from July 1, 1945, to June 30, 1946.

TABLE I—SUMMARY OF RECEIPTS AND EXPENDITURES OF COMMITTEE FUNDS.

Balance as of June 30, 1945 shown in 1945 Report			$8 622.40
	RECEIPTS		
1945			
September	Interest on funds (10/1 44 to 9/30/45)	36.07	
November	Engineering Foundation	1 000.00	
1946			
January	Engineering Foundation	1 000.00	
April	Engineering Foundation	1 000.00	
	Total Receipts		3 036.07
			$11 658.47
	EXPENDITURES		
1945			
September	Printing Piping Papers	40.00	
December	Printing Piping Papers	62.92	
1946			
June	Battelle Memorial Institute (Project No. 18)	54.86	
	Total Expenditures		157.78
Balance on hand, June 30, 1946			$11 500.69

Organization and Membership:

The committee records with deep regret the passing away on April 21, 1946, of H. J. Kerr, vice-chairman of the committee. Mr. Kerr had joined the committee in February 1930, and had been one of its most active members during the years since that time. He became vice-chairman in 1938. His knowledge of engineering in his chosen field, and of the application of materials in critical service, and his mature guidance at all times, were extremely valuable to the work of the committee.

There have been several changes in membership during the past year. Russell Franks of the Electro Metallurgical Corporation has been appointed as a new member. Howard C. Cross, of Battelle Memorial Institute, long active in the work of the committee, has been appointed to membership in place of H. W. Gillett, who has resigned. J. G. Thompson is now representing the National Bureau of Standards in the place of H. S. Rawdon.

Research Projects:

Project No. 29 on Studies on Susceptibility of Casting Steels to Graphitization (J. J. Kanter, chairman).—A report on this project is appended hereto.

Respectfully submitted on behalf of the Joint Research Committee,

N. L. MOCHEL,
Chairman.

J. W. BOLTON,
Secretary.

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[4] See p. 970.

[5] See p. 870.

[6] See p. 902.

APPENDIX

STUDIES ON SUSCEPTIBILITY OF CASTING STEELS TO GRAPHITIZATION

(PROJECT No. 29)

PREPARED BY J. J. KANTER[1] AND E. A. STICHA[1]

A program of tests was adopted by the Manufacturers Standardization Society of the Valve and Fitting Industry in cooperation with Project No. 29 to obtain information on the graphitization of steel castings. Investigation of certain steels of types covered by A.S.T.M. Tentative Specifications for Alloy - Steel Castings Suitable for Fusion Welding for High-Temperature Service (A 217)[2] have been under study for more than a year. The test castings and investigation services in the program were contributed by the following manufacturers: Chapman Valve Mfg. Co., Crane Co., Edward Valves, Inc., Lunkenheimer Co., Wm. Powell Co., Reading-Pratt & Cady Div. of Am. Chain and Cable Co., Inc., and Walworth Co.

A previous progress report concerned with the study of the castings was presented at the session on Graphitization of Steel Piping held at the Annual Meeting of The American Society of Mechanical Engineers on November 28, 1945, under the auspices of the Joint Committee.[3] This report described the test castings, the preparation of weld-bead specimens for aging, and the materials involved. Aging periods up to 6000 hr. were reported to have produced graphitization in aluminum-killed carbon-molybdenum steel melted by various electric processes. At the time of this previous report, no graphitization had been observed for chromium-molybdenum steel samples containing chromium contents in the range of 0.43 to 0.70 per cent. It was reported that carbon - molybdenum samples normalized after deposition of weld bead had developed graphite in the heat-affected zone. The data had not indicated any preference among acid arc, basic arc, and induction melting as far as graphitization is concerned.

At the present time, further observations upon these cast steel specimens are available for aging periods up to 10,000 hr. In the accompanying Tables I and II are given the analyses, results of McQuaid-Ehn structure, and appraisals on the indications of graphite after various aging periods at 1025 F. A few photomicrographs (Fig. 1) are included to illustrate graphite identified in some of the chromium-molybdenum steel samples.

For the purpose of discussing the current indications of these data, the cast steels represented may be treated in several groups as follows:

1. Carbon-molybdenum steel, treated with 2 lb. of aluminum per ton containing chromium ranging from 0.01 to 0.07 per cent.

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[1] Materials Research Engineer and Assistant Materials Research Engineer, respectively, Crane Co., Chicago, Ill.

[2] 1944 Book of A.S.T.M. Standards, Part I, p. 1281.

[3] *Transactions*, Am. Soc. Mechanical Engrs., Vol. 68, No. 6, pp. 581 to 587 (1946).

2. Carbon-molybdenum steel, treated with 2 lb. of aluminum per ton containing chromium ranging from 0.17 to 0.35 per cent.

3. Chromium - molybdenum steel, treated with 2 lb. of aluminum per ton containing chromium ranging from 0.43 to 0.70 per cent.

4. Carbon - molybdenum steel, treated with ½ lb. of aluminum per ton.

5. Carbon - molybdenum steel, treated with silicon, only.

6. Carbon - molybdenum steel, treated with 2 lb. of aluminum per ton, normalized and drawn after deposition of weld bead.

TABLE I. CHEMICAL ANALYSES OF CAST CARBON-MOLYBDENUM STEELS FOR PROJECT NO. 29 GRAPHITIZATION SUSCEPTIBILITY TESTS.

Aluminum Additions
Steels D, G. 0
Steels A, E. .... ½ lb. per ton
Steels B, C, F. .... 2 lb. per ton

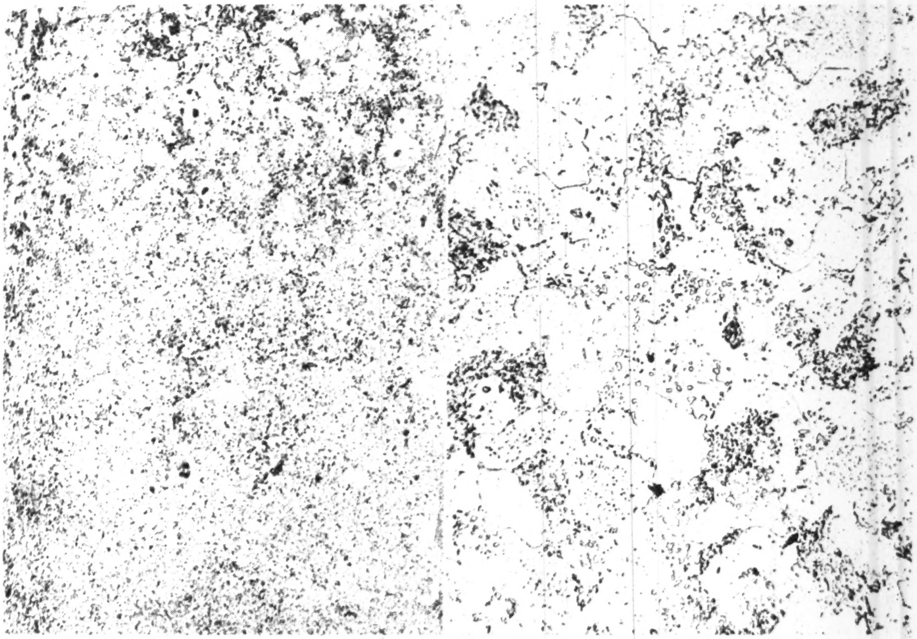
Designation	Si	Mn	S	P	C	Ni	Cr	Mo	Cu	W	V	Al Total	Al Acid Soluble	Al_2O_3	Graphite	Other Additions
1A	0.32	0.66	0.038	0.037	0.25		0.03	0.40				0.009				5 lb. per ton Carbortam (16 per cent Ti)
1B	0.30	0.70	0.034	0.041	0.25		0.04	0.40				0.055				
1C	0.42	0.60	0.034	0.037	0.21		0.61	0.45								1 lb. per ton Ca Si
2A	0.28	0.57	0.028	0.031	0.22	0.08	0.05	0.43	0.04			0.014		0.0223	0.021	
2B	0.29	0.58	0.028	0.030	0.21	0.10	0.01	0.44	0.04			0.063		0.0108	0.013	
2C	0.35	0.72	0.031	0.033	0.26	0.17	0.44	0.43	0.09			0.069		0.0186	0.029	
2E	0.34	0.72	0.031	0.033	0.27	0.09	0.43	0.43	0.09			0.016		0.0211	0.043	
3A	0.48	0.67			0.23		0.10	0.53								
	0.39	0.62			0.20		0.05	0.57				0.03				1.1 lb. Al per ton (Total)
3B	0.48	0.67			0.23		0.10	0.53								
	0.40	0.67			0.20		0.07	0.57				0.10				
3C	0.36	0.62			0.17		0.54	0.54								
	0.40	0.64			0.13		0.59	0.57				0.11				
3D	0.48	0.67			0.17		0.10	0.53								
	0.35	0.64			0.20		0.05	0.57				0.02				0.4 lb. Al per ton
3F	0.48	0.67			0.17		0.10	0.53								
	0.39	0.68			0.22		0.09	0.57				0.15				
4A	0.30	0.58	0.037	0.024	0.20	0.11	0.07	0.43	0.05							
4B	0.30	0.58	0.037	0.024	0.20	0.11	0.07	0.43	0.05							
4C	0.41	0.68	0.034	0.022	0.24	0.05	0.43	0.43	0.07							
4D	0.30	0.58	0.037	0.024	0.20	0.11	0.07	0.43	0.05							
4E	0.41	0.68	0.034	0.022	0.24	0.05	0.43	0.43	0.07							
4G	0.41	0.68	0.034	0.022	0.24	0.05	0.43	0.43	0.07							
5A	0.31	0.58	0.035	0.027	0.25	0.046	0.033	0.54	0.056	Nil						
5B	0.31	0.58	0.035	0.027	0.25	0.046	0.033	0.54	0.050	Nil						
5C	0.27	0.60	0.037	0.023	0.20	0.052	0.56	0.54	0.055	Nil						
5D	0.31	0.58	0.035	0.027	0.25	0.046	0.033	0.54	0.050	Nil						
5E	0.27	0.60	0.037	0.023	0.20	0.052	0.56	0.54	0.055	Nil						
5G	0.27	0.60	0.037	0.023	0.20	0.052	0.56	0.54	0.055	Nil						
6B	0.40	0.63	0.006	0.033	0.24	0.29	0.25	0.54	0.10		0.02	0.05	0.050	0.008		
6C	0.32	0.70	0.038	0.022	0.17	0.31	0.57	0.48	0.12		0.06	0.052	0.040	0.012		
6D	0.64	0.62	0.006	0.032	0.24	0.29	0.25	0.54	0.10		0.02	0.029	0.019	0.910		
7A	0.44	0.70	0.015	0.024	0.26	0.0	0.03	0.49	0.36		0					
7B	0.43	0.70	0.015	0.024	0.26	0.12	0.05	0.46	0.33		0					
7C	0.44	0.66	0.015	0.024	0.26	0.12	0.52	0.47	0.33		0					
6BI	0.40	0.46	0.026	0.023	0.19	0.25	0.34	0.52	0.13		0.01	0.046	0.033	0.013		
6CI	0.39	0.47	0.026	0.023	0.19	0.25	0.70	0.53	0.13		0.02	0.047	0.032	0.015		
6DI	0.61	0.46	0.026	0.023	0.19	0.25	0.34	0.52	0.13		0.01	0.024	0.018	0.009		
6DIsI	0.46	0.69	0.026	0.032	0.24	0.26	0.34	0.54	0.10		0.02	0.022	0.012	0.010		
7AI	0.37	0.68	0.027	0.023	0.24	0	0.03	0.52	0.16		0					
7BI	0.38	0.66	0.028	0.025	0.23	0	0.03	0.52	0.08		0					
7CI	0.40	0.66	0.030	0.023	0.23	0	0.50	0.52	0.07		0					
7DI	0.47	0.60	0.032	0.023	0.20	0	0.05	0.50	0.13		0					
DI5	0.36	0.64	0.006	0.033	0.24	0.29	0.27	0.54	0.09		0.02	0.029	0.019	0.010		
[illegible]	0.37	0.62			0.12		0.61	0.57				0.16				3 lb. Al per ton
C	0.38	0.66	0.028	0.020	0.23		0.48	0.51								
[illegible]	0.38	0.66	0.028	0.020	0.23		0.35	0.51								2 lb. Al per ton
B	0.40	0.62	0.032	0.016	0.22		0.17	0.47								
[illegible]	0.19	0.68	0.010	0.011	0.35	Tr.	0.34	0.60	Tr.							4½ lb. Al per ton
X	0.36	0.55	0.024	0.022	0.21		0.29	0.50								2½ lb. Al per ton

NOTE.—These analyses furnished by the various co-operators.

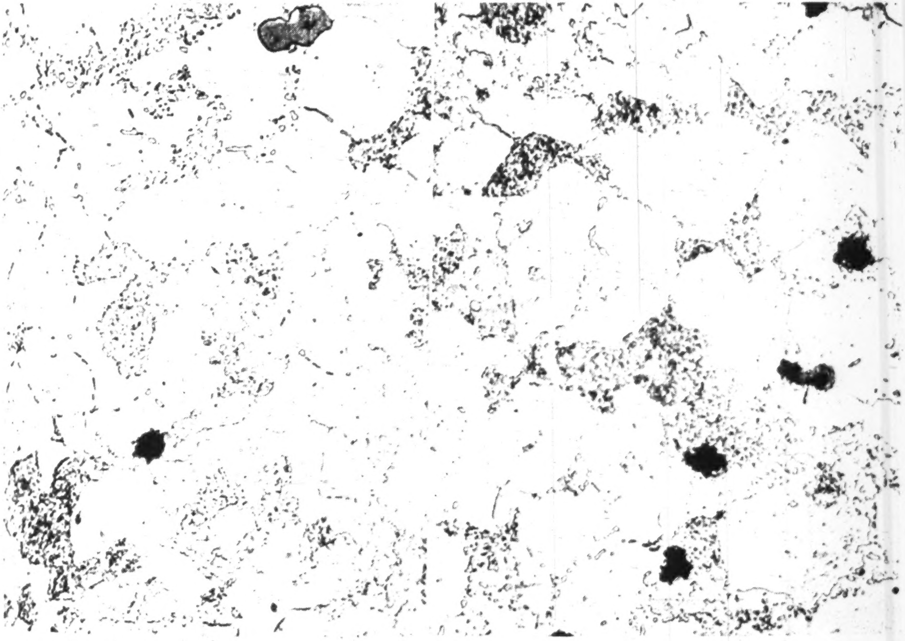
From a consideration of the data for groups 1, 2, and 3, an attempt has been made to appraise the effectiveness of chromium additions to carbon-molybdenum cast steels treated with 2 lb. of aluminum per ton. In Table II are listed the appraisals of the individual micro-examinations of groups 1, 2, and 3 samples at various aging intervals from 1000 to 10,000 hr. Estimates have been made of the portion of the carbon at the critical zone which has transformed to

TABLE II.—ESTIMATED PERCENTAGE OF CARBON TRANSFORMED TO GRAPHITE FOR WC1 AND WC3 CAST STEELS AGED AT 1025 F.

Mark	Cr, per cent	Grain Size	Aged 1000 hr.	Aged 1500 hr.	Aged 2000 hr.	Aged 3000 hr.	Aged 4000 hr.	Aged 4500 hr.	Aged 5000 hr.	Aged 6000 hr.	Aged 8000 hr.	Aged 9000 hr.	Aged 10 000 hr.
GROUP 1—WC1-DEOXIDIZED WITH 2 LB. OF ALUMINUM PER TON													
1B	0.04	Fine	0				0	0			10		
2B	0.01		0		5	50	50		75			80	
4B	0.07	6 to 7	10				25		50		75		
5B	0.03		0		1	3	50		75			75	
7B	0.05	8		10		25		50		75			
7BI	0.03	8 to 9		35				75		100			
Average	0.04	8	3	10	13	25	38	48	58	65	75	80	
GROUP 2—WC1 PLUS ¼ PER CENT CHROME AND DEOXIDIZED WITH 2 LB. ALUMINUM PER TON													
6B	0.25	6 to 8	0		0	0	0		2			20	
6BI	0.34	6 to 8				0				0			
7B	0.17			0		0		0		0			0
7✱	0.34	8		0		0		0		0			0
7C′	0.35	8 to 9		0						10			20
7×	0.29	8								10			
Average	0.29	8	0	0	0	0	0	0	2	4		8	11
GROUP 3—WC3-DEOXIDIZED WITH 2 LB. ALUMINUM PER TON													
1C	0.61	Fine	0				0		0		0		
2C	0.44		0		0	0	1		2			0	
4C	0.43	7	0				0		0		0		
5C	0.56		0		0	0	0		1			0	
6C	0.57	8 to 9	0		0	10	0		2			2	
7C	0.52	8 to 9		0		0		0		0			10
6CI	0.70	7 to 9				0				0			
7CI	0.50	8 to 9		0		0		0		0			
7Y	0.48	9		0		0		0		0			0
Average	0.53	8 to 9	0	0	0	1	1	1	1	1	1	1	2
GROUP 4—WC1-DEOXIDIZED WITH ½ LB. ALUMINUM PER TON													
1A	0.03		0				0		0		0		
2A	0.05		0		0	0	0		0			50	
4A	0.07	6	0				0		0		24		
5A	0.03		0		0	0	0		0			2	
7A	0.03	7 to 8		0		0				20			
7AI	0.03	7		0				30		50			
5E	0.56		1		0	0	0		5			0	
Average	0.09	7	0	0	0	0	0	5	10	13	22	27	
GROUP 5—WC1-DEOXIDIZED WITH SILICON ONLY													
7D	0.05	3 to 5		0		0		0		0			
6DIs	0.34	3 to 5	0		1	0	2		10			0	
6DI	0.34	3 to 5				0				0			
6DIsI	0.34	3 to 5	0							0			
4G	0.43	2 to 3	0				0		0	0			
Average	0.30	4	0	0	0	0	0	0	0	0		0	
GROUP 6—WC1-DEOXIDIZED WITH 2 LB. ALUMINUM PER TON, NORMALIZED AND DRAWN AFTER WELDING													
1BH	0.04		0				0		5		10		
2BH	0.01		0		0	0	0		25			25	
4BH	0.07		0				0		10		20		
5BH	0.03		0		0	0	2		10			50	
6BH	0.25		0		0	0	1		15			15	
7BH	0.05			0		0		0		10			15
7BIH	0.03			5				15		20			30
Average	0.07		0	1	1	1	2	7	12	16	20	25	29



Contact Zone (× 100) Contact Zone (× 500)



1/16 in. from Contact Zone, Base Metal Side (× 500) ⅛ in. from Contact Zone, Base Metal Side (× 500)

FIG. 1.—Photomicographs Showing Graphite in a Cast 0.5 per cent Chromium–0.5 per cent Molybdenum Steel After 10,000 hr. at 1025 F.

graphite. The graphitized portion is expressed in percentage and has been averaged for each group. Inspection of the cases in group 1 shows that at the end of 10,000 hr. of aging at 1025 F., graphitization has advanced to a degree of unmistakable consequence. Groups 2 and 3 began to show isolated nodules of graphite after 4000 hr. of aging at 1025 F. Obviously, these latter two groups are much more resistant to graphitization than group 1.

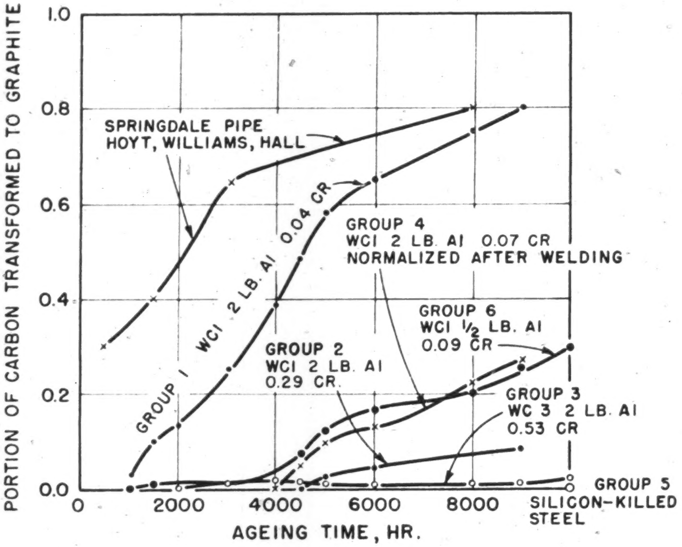


FIG. 2.—Reaction Curves for Graphitization at 1025 F.

In Fig. 2, the average percentage of carbon transformed to graphite is shown *versus* the aging time. A similar set of data for the Springdale pipe by Hoyt, Williams, and Hall[4] are shown in this figure for comparison with the cast steels. It is quite apparent that cast WC1 and WC3 steels are considerably more resistant to graphitization at 1025 F. than the Springdale pipe.

The group 1 steels present a reaction curve which tends to confirm the conception that graphitization proceeds according to a nucleation and diffusion process. The curves for groups 2 and 3 are not as well defined as that for group 1, the segments determined in 10,000 hr. of aging seeming to represent scarcely more than the nucleation stage and suggest that much longer aging periods will be required to determine accurately the upward sweep of these chromium-bearing steels.

The data represented in Fig. 1 unquestionably demonstrate that a small chromium content retards the rate at which graphite forms in cast steel containing 0.5 per cent of molybdenum and treated with 2 lb. of aluminum per ton. A chromium content of 0.5 per cent, however, cannot be considered as demonstrated as sufficient to inhibit adequately the formation of consequential amounts of graphite at 1025 F. if the indicated trends be extrapolated toward long periods of time.

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[4] Summary Report on the Joint E. E. I.-A.E.I.C. Investigation of Graphitization of Piping, November, 1945 presented at A.S.M.E. Session on Graphitization of Stee Piping.

The curve for group 4 steels, treated with $\frac{1}{2}$ lb. of aluminum per ton, are seen by Fig. 1 practically to coincide with that for group 6 steels which were normalized and drawn after welding. Furthermore, the curves are seen to lie intermediately to the curves for WC1, treated with 2 lb. of aluminum per ton and WC1, treated with 2 lb. of aluminum per ton plus 0.25 per cent chromium. Estimate of the "half graphite" time for groups 4 and 6 indicates that the effectiveness of either treatment increases the resistance about the same as an addition of 0.15 per cent of chromium. Thus, while such measures seem to be helpful in establishing greater stability, they do not seem of themselves sufficient to gain adequate serviceability.

The group 5 results shown in Table II for the steels deoxidized with silicon only seem to be showing exceptional stability. None of the samples in this group has exhibited as yet any persistent tendency to develop graphite. Unfortunately, however, as was pointed out in the previous progress report, these castings are not free of "pinholes."

Although final conclusions may not as yet be drawn from the data presented in Fig. 1, the actual findings indicate the importance of extending the studies to include certain further steels not embraced in the original program of tests. At the time that this program of aging tests was undertaken, it was anticipated that the addition of 0.5 per cent of chromium to the WC1 composition might furnish a readily weldable casting steel suitable for the requirements of steam power plant piping to operate in the range of temperature from 900 to 1000 F. Now that test data are suggesting that another year or two of aging may dispel such an expectation, it becomes highly urgent that the stability of more highly alloyed casting steels be appraised. Some tests of such steel have already been undertaken by the MSS group, and others are to be prepared. The more highly alloyed compositions particularly under consideration are grade WC1 of Specifications A 217,[2] plus 1 per cent of chromium, grade WC4 (Ni-Cr-Mo) of A 217, and grade C3A of A 157[5] containing 1.5 to 2.25 per cent chromium, 0.5 per cent molybdenum, but restricted to a carbon maximum compatible with acceptable welding characteristics.

If these steels prove to present a greater order of stability against graphitization than the present grade WC3 (0.5 per cent Mo-0.5 per cent Cr), aging tests upon them at 1025 F. cannot be expected to afford impressive information on the prospects of 200,000 hr. of serviceability for several years to come. It is, therefore, now under consideration to undertake the additional aging of some of these new steels of richer alloy content at 1100 F. According to the temperature relationship presented by Hoyt, *et al*,[4] indications at 1100 F. may be expected to develop in about one third of the time required at 1025 F. This would mean that 1100 F. on the higher alloys may not be expected to yield stability criteria of interest for at least two years. Samples of the present WC3 composition under test are also included in the 1100 F. aging tests to afford a means of correlation between the more highly alloyed steels and the 1025 F. data. Significant indications for WC3 samples at 1100 F. should be available in about a year if the temperature relationship established at Battelle for carbon-molybdenum pipe also holds valid for the cast chromium-molybdenum steels.

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[5] Standard Specifications for Alloy-Steel Castings for Valves, Flanges, and Fittings for High-Temperature Service (A 157 – 44), 1944 Book of A.S.T.M. Standards, Part I, p. 295.

REPORT OF COMMITTEE B-1*

ON

WIRES FOR ELECTRICAL CONDUCTORS

Committee B-1 on Wires for Electrical Conductors held two meetings during the year in New York, N. Y., on February 15, 1946, and May 10, 1946.

The committee now consists of 49 members, of whom 22 are classified as producers, 19 as consumers, and 8 as general interest members.

At the February, 1946, meeting of the committee, the election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, J. H. Foote.
Vice-Chairman, W. R. Hibbard.
Secretary, E. H. Kendall.

In addition to the recommendations affecting standards reported below, a task group has made progress toward the formulation of methods of tests for determining the actual areas of cross-section of finished stranded conductors. This is a matter involving practical difficulties which has evaded solution for a number of years.

Another task group is studying the possible conversion of the Tentative Method of Test for Resistivity of Copper and Copper-Alloy Electrical Conductors (B 193 – 45 T) to a conductivity basis since the low resistivities associated with electrical "conductors" might preferably be expressed as conductivities. This work is under review by a joint committee representing those commiteees of the Society concerned with the problem.

Change in Scope of Committee B-1.—During the year authorization was given by the Society for the change in scope of this committee from "Copper and Copper-Alloy Wires for Electrical Conductors" to read "Specifications and Methods of Test for Uninsulated Electrical Conductors Composed of Wires in Various Fabrications." This enlargement in scope will add considerably to the work of this committee and has made it desirable to adopt formal regulations governing the committee. These regulations will be published separately and will be available from the secretary of the committee. They provide for a more formal organization and a more extensive system of subcommittees than has been heretofore employed by this committee. The membership will be augmented to include representation for other conductor materials than copper and for combinations of materials. It is expected that the enlarged program of work will be organized and under way some time in the coming fall or winter months.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revisions in five standards and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society:

Standard Specifications for Bronze Trolley Wire (**B 9 – 39**):[1]

Section 3 (*b*).—Change the first sentence to read as follows: "Tests on a specimen of round wire containing a joint shall show at least 95 per cent of the tensile strength specified in Table I."

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] 1944 Book of A.S.T.M. Standards, Part I.

Section 6 (b).—Change the first sentence to read as follows: "Tests on a specimen of grooved wire containing a joint shall show at least 95 per cent of the tensile strength specified in Table II."

Standard Specifications for Copper Trolley Wire (**B 47 – 39**):[1]

Section 3 (b).—Change the first sentence to read as follows: "Tests on a specimen of round wire containing a joint shall show at least 95 per cent of the tensile strength specified in Paragraph (*a*)."

Section 6 (b).—Change the first sentence to read as follows: "Tests on a specimen of grooved wire containing a joint shall show at least 95 per cent of the tensile strength specified in Paragraph (*a*)."

Standard Specifications for Figure-9 Deep-Section Grooved and Figure-8 Copper Trolley Wire for Industrial Haulage (**B 116 – 40**):[1]

Section 3 (c).—Change the first sentence to read as follows: "Tests on a specimen of wire containing a joint shall show at least 95 per cent of the tensile strength specified in Table I or Table II, as may be applicable."

The recommended revisions in the above three specifications are of similar import and are designed to make definite the required tensile values at joints in the wire. Thus the revision requires that the strength of the joint be not less than 95 per cent of definite tabular values rather than 95 per cent of the tested strength of samples of the wire.

Standard Specifications for Concentric-Lay-Stranded Copper Conductors, Hard, Medium-Hard, or Soft (**B 8 – 44**):[1]

Table I.—Delete the requirement of "50 ft." for the 12 wire strand in class A since the 12 wire strand is no longer included in class A.

Table II.—Change the number of wires of class A stranding for sizes 0000 and 000 from "12" to read "7" and change the diameter of wires of class A stranding for size 0000 from "132.8" to "173.9" and for size 000 from "118.3" to "154.8"; also delete footnote *a* and its reference.

This recommended revision will eliminate an option not found to be necessary in commercial practice and will also remove undesirable conflicts in the application of the specifications.

Table III.—Delete the lines showing 0000, 12 wire construction and 000, 12 wire construction.

Table IV.—Add asterisks before the 0000, 3-wire constructions for Nos. 1, 2, 3, and 4.

Standard Specifications for Tinned Soft or Annealed Copper Wire for Electrical Conductors (**B 33 – 45**):[2]

Section 8 (b).—In the third line, insert the abbreviation "c.p." (for the trade designation "chemically pure"), between the words "sulfide" and "crystals." In the fifth line, delete the word "about" from the phrase "about 250 g." At the end of this paragraph add the reference "(Note 7)."

Explanatory Notes.—Add a new Note 7 to read as follows:

NOTE 7.—It is important that the polysulfide solution be of proper composition and strength at the time of test. A solution which is not saturated with sulfur or which has been made from decomposed crystals may give negative results. Therefore, the requirement that the solution be tested by observing its blackening effect on a bright copper wire is significant. Significant also is the requirement that the solution be saturated with sulfur by allowing the solution to stand at least 24 hr. after preparation. Attention is called also to the necessity for the use of sodium sulfide which has not deteriorated through exposure to air; and if exposure has occurred, the crystals should be tested for purity. The "Standard Reagents Tests" of the American Chemical Society are useful in this connection.

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[2] 1945 Supplement to Book of A.S.T.M. Standards, Part I.

This clarification of the sodium polysulfide solution preparation and its significance in the testing procedure has been found necessary through practical experience in the application of these specifications within the past few years.

TABLE I. ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
REVISION OF STANDARDS, IMMEDIATE ADOPTION			
Spec. for Bronze Trolley Wire (B 9 – 39)	31	0	14
Spec. for Copper Trolley Wire (B 47 – 39)	32	0	13
Spec. for Figure-9 Deep-Section Grooved and Figure-8 Copper Trolley Wire for Industrial Haulage (B 116 – 40)	31	0	14
Spec. for Concentric-Lay-Stranded Copper Conductors, Hard, Medium-Hard, or Soft (B 8 – 44)	29	0	12
Spec. for Tinned Soft or Annealed Copper Wire for Electrical Conductors (B 33 – 45)	39	0	6

REAFFIRMATION OF STANDARD

The committee recommends that the Standard Specifications for Hard-Drawn Copper Alloy Wires for Electrical Conductors (B 105 – 39) which has been published six years without revision be reaffirmed and continued as standard without change.

The recommendations in this report have been submitted to letter ballot of the committee, which consists of 49 members, with the results shown in Table I.

This report has been submitted to letter ballot of the committee, which consists of 49 members; 45 members returned their ballots, of whom 44 voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. H. FOOTE,
Chairman.

E. H. KENDALL,
Secretary.

REPORT OF COMMITTEE B-2*

ON

NON-FERROUS METALS AND ALLOYS

Committee B-2 on Non-Ferrous Metals and Alloys because of travel restrictions and in line with Government request to limit meetings has not held a meeting of the committee as a whole since June 29, 1944. During this period, business of the committee and subcommittees has been conducted by correspondence. Subcommittees assigned work to task groups, and the reports of these groups were submitted by letter and letter ballot to the subcommittees. Final action of the committee as a whole in these matters was as usual by letter ballot, which letter ballot was accompanied by letter of explanation.

The present officers of Committee B-2 have been re-elected for the ensuing term of two years.

Proposed Tentative Specifications for Fire-Refined Copper for Wrought Alloys[1] have been prepared by Subcommittee I to supersede Emergency Specifications for Fire-Refined Copper for Wrought Products and Alloys (ES – 7).[1] The withdrawal of Standard Specifications for Fire-Refined Copper Other Than Lake (B 72 – 33) is being considered because of apparent lack of necessity. It is expected that Committee B-2 will be able to act on both of these proposals prior to publication of the 1946 Book of A.S.T.M. Standards.

Revisions of the Standard Specifications for White Metal Bearing Alloys (B 23 – 26) and the Tentative Specifications for Soft Solder Alloys (B 32 – 40 T) are in preparation in Subcommittee III.[1] The revised specifications are to be published as tentative and will supersede the present standard specifications and their emergency alternate provisions. The task group is completing its work and plans to send recommendations to the subcommittee promptly. It is expected that Committee B-2 will be able to act on both proposed tentative specifications prior to publication of the 1946 Book of Standards.

Acceptance of the before mentioned three proposed tentative specifications would remove the last of war-time emergency alternate provisions and emergency specifications under jurisdiction of Committee B-2.

The Advisory Committee held a meeting in New York, N.Y., on March 25, 1946. Mr. Sidney Rolle was appointed chairman, *pro tem*, of Subcommittee II on Refined Lead, Tin, Antimony and Bismuth. Changes in membership were approved and business before the subcommittees discussed. The chairmen of subcommittees outlined their plans to complete any work relating to standards in order that revised specifications may appear in the new Book of A.S.T.M. Standards to be issued this year.

Committee B-5 on Copper and Copper Alloys, Cast and Wrought, had suggested that the work of Subcommittee I be transferred to its jurisdiction; that is, Committee B-5 wishes to extend its scope to cover not only the fabrication of copper and copper alloys but to include jurisdiction over the production of copper.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] See Editorial Note, p. 193.

It was the expression of the Advisory Committee that unless arguments advanced by Committee B-5 were cogent, the work of Subcommittee I should continue in Committee B-2. It was decided to submit the question to the members of Subcommittee I and their opinion would guide the action of the members of the Advisory Committee before the Society. At a meeting of Subcommittee I, on April 19, 1946, in New York, the subcommittee was definitely not in favor of the transfer of its functions to Committee B-5.

There had been an inquiry by a consumer of lead as to sampling procedure. The Chairman of Subcommittee II was furnished with copies of the correspondence. The Subcommittee will recommend whether any revision of the sampling procedure is necessary.

It was decided to determine whether at this time it is necessary or desirable to propose specifications for tin and specifications for antimony.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption that in the Standard Specifications for Slab Zinc (Spelter) (B 6 – 37) the requirements for chemical composition of special high-grade zinc be revised to read as follows:

Special High-Grade Zinc	
Lead, max., per cent	0.006
Iron, max., per cent	0.005
Cadmium, max., per cent	0.004
Aluminum, max., per cent	none
Sum of lead, iron, and cadmium, max., per cent	0.010

NOTE.—Analysis shall not regularly be made for tin but when used for die castings, if found by the purchaser, tin shall not exceed 0.003 per cent. Greater amounts may constitute cause for rejection.

This recommendation has been submitted to letter ballot of the committee, which consists of 134 members; 77 members returned their ballots, of whom 49 have voted affirmatively, 1 negatively, and 27 members marked their ballots "not voting."

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Refined Copper (J. L. Christie, chairman) has prepared Proposed Tentative Specifications for Fire-Refined Copper for Wrought Alloys to supersede Emergency Specifications for Fire-Refined Copper for Wrought Products and Alloys ES-7. The subcommittee is considering recommending the withdrawal of Standard Specifications for Fire-Refined Copper Other than Lake (B 72 – 33) because of lack of necessity. The subcommittee in its work on preparing Classification of Coppers requests that the Coordinating Committee on Non-Ferrous Metals and Alloys determine if such a classification is needed and if so, to suggest how the need could best be filled.

Subcommittee II on Refined Lead, Tin, Antimony and Bismuth (Sidney Rolle, chairman) is considering the sampling procedure in the Standard Specifications for Pig Lead (B 29 – 43) to determine whether any changes are necessary. The subcommittee is also considering whether it is necessary or desirable to prepare tentative specifications for tin and tentative specifications for antimony.

Subcommittee III on White Metal Alloys (G. H. Clamer, chairman) through its task group has under completion the revised specifications for white metal bearing alloys and for soft solder alloys as mentioned earlier in this report. The task group in preparing these specifications sought to include the soft solders and white metal bearing alloys designated by the Society of Automotive Engineers and Federal Specifications.

Subcommittee IV on Refined Zinc and Wrought Zinc (E. H. Bunce, chairman) has reaffirmed the Standard Specifications for Rolled Zinc (B 69 – 39).

Committee B-2 has accepted the subcommittee's proposed revision of Standard Specifications B 6 as given earlier in this report.

A request for specifications for zinc-base sheets for mechanically grained lithoplates has been received and the subcommittee is considering whether specifications are necessary or desirable.

Subcommittee V on Precious Metals and Alloys (R. H. Leach, chairman) has reviewed and reaffirmed the Standard Specifications for Silver Solder (B 73 – 29).

Subcommittee VI on Coated Metals.—A chairman is to be appointed for this subcommittee in the near future and the subcommittee will review the Standard Specifications for Lead-Coated Copper Sheet (B 101 – 40) and consider any other work which may come before the subcommittee.

Subcommittee VII on Refined Nickel and High-Nickel Alloys, Cast And Wrought (O. B. J. Fraser, chairman) is reviewing the specifications under its jurisdiction for the purpose of determining whether any revisions are necessary.

Subcommittee VIII on Miscellaneous Refined Metals and Alloys (E. E. Schumacher, chairman) has requested that the Coordinating Committee on Non-Ferrous Metals and Alloys decide what committee has jurisdiction over Standard Specifications for Brazing Solder (B 64 – 43).

This report has been submitted to letter ballot of the committee, which consists of 134 members; 93 members returned their ballots, of whom 78 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

E. E. THUM,
Chairman.

G. HOWARD LEFEVRE,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Specifications for:
- Fire-Refined Copper for Wrought Alloys (B 216 – 46 T), formerly Emergency Specifications ES-7, and
- White Metal Bearing Alloys (B 23 – 46 T).

Revision of Tentative Specifications for:
- Soft Solder Metal (B 32 – 46 T).

Withdrawal of Standard Specifications for:
- White Metal Bearing Alloys (Known Commercially as "Babbitt Metal") (B 23 – 26).

These recommendations were accepted by the Standards Committee on November 25, 1946, and the new and revised tentatives appear in the 1946 Book of A.S.T.M. Standards, Part I-B.

At the same time, the Standards Committee accepted the recommendation of Committee B-2 that the following emergency provisions be withdrawn:

Emergency Specifications for Fire-Refined Copper for Wrought Products and Alloys (ES – 7),

Emergency Alternate Provisions in the Standard Specifications for White Metal Bearing Alloys (Known Commercially as "Babbitt Metal") (B 23 – 26), and

Emergency Alternate Provisions in the Tentative Specifications for Soft Solder Metal (B 32 – 40 T).

REPORT OF COMMITTEE B-3*

ON

CORROSION OF NON-FERROUS METALS AND ALLOYS

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys held a meeting in Pittsburgh, Pa., on February 26, 1946.

The committee consists of 84 members, of whom 71 are voting members; 32 are classified as producers, 29 as consumers, and 10 as general interest members.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, H. S. Rawdon.
Vice-Chairman, A. C. Halferdahl.
Secretary, A. W. Tracy.

Editorial Change in Tentative Method

The committee voted to continue as tentative the Method of Total Immersion Corrosion Testing of Non-Ferrous Metals (B 185 – 43 T) with the following editorial change:

Section 8(a).—Add the following sentence to the end of this paragraph: "Loss in weight measurements will not show the true corrosion rate if metals are severely pitted or selectively corroded."

Activities of Subcommittees

Subcommittee I on Total Immersion Test (A. W. Tracy, chairman).—The Recommended Practice for Plant Corrosion Tests (A 224 – 41) developed by Committee A-10 on Iron-Chromium, Iron-Chromium Nickel and Related Alloys, has been reviewed by the subcommittee and changes have been made to make it applicable to the testing of non-ferrous metals and alloys. On the recommendation of this subcommittee, Committee B-3 voted to recommend to Committee A-10 that one method be written to cover the testing of all metals.

Subcommittee III on Spray Test (C. E. Heussner, chairman).—This subcommittee through its Subgroup 3 has been engaged in studying the effect of the important factors in the Tentative Method of Salt Spray (Fog) Testing (B 117 – 44 T). It has been found that between the critical air pressure limits of 13 and 16 psi., pressure variations of plus or minus 0.5 psi. will significantly affect the corrosiveness of the fog. Investigation has shown that with air pressure varying plus or minus 0.5 psi., each particular type of nozzle has a critical pressure at which point the corrosive effect of the fog is abnormally severe. This critical pressure has been found between 13 and 16 psi. It was recommended the pressure be maintained constant within 0.2 psi.; this might be accomplished with two diaphragm valves in series in the air line.

Studies have shown that baffling, nozzle distance, and settling rate are adequately covered by the present method with respect to sheet zinc and cold-rolled steel. Further work on the settling rate is planned on the following metals:

S.A.E. 1010 steel,
S.A.E. 1010 steel, plus 0.0005 in. nickel,
S.A.E. 1010 steel, plus 0.0005 in. zinc,
S.A.E. 1010 steel, plus 0.0002 in. cadmium,
S.A.E. 1010 steel, phosphated and painted,
Zinc plus chromate film,

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

24ST Aluminum,
24ST Aluminum, chromic acid anodized,
AM-C52S Magnesium, and
AM-C52S Magnesium, chrome pickled.

As regards the effect of pH, Monel tanks for salt solution and phenol-formaldehyde resins in the chamber have caused considerable variation in the pH of fog. If the pH of salt solutions is adjusted at room temperature, the pH of the fog will vary considerably from the range of 6.5 to 7.2 now specified. This range is narrower than necessary for iron or zinc; however, a higher pH of salt fog is more severe on paint coatings. It has been found that the salt solution should be heated for 48 hr. at 95 F., or boiled for 1 min. before making pH adjustments and that the pH of the collected fog should be checked frequently. Further work on the effect of pH on corrosion rate is being considered.

Regarding the effect of heavy-metal impurities on light metal samples, work is being done to determine the limits of heavy metals, copper and nickel, permissible in salt spray solutions in spray boxes testing light metals.

Subcommittee V on Statistical Analysis and Planning of Corrosion Testing (W. E. Campbell, chairman).—The first objective of the subcommittee has been achieved in the presentation at the 1946 Spring Meeting of a paper commenting, from a statistical viewpoint, on the results of the Subcommittee VI atmospheric corrosion tests. In this paper recommendations are made for the conduction of future tests.

A meeting of Subcommittee V was held at the time of the Spring Meeting. It was agreed that, in order to function with maximum efficiency, Subcommittee V should be given full details of the scope and objectives of new testing programs as soon as possible after the program has been agreed upon.

Subcommittee VI on Atmospheric Corrosion (W. H. Finkeldey, chairman).—The atmospheric exposure tests on nonferrous metals and alloys which the subcommittee started in 1932 are still continuing. The fifth and last removal of test specimens from the exposure racks will probably not be made for another 5 or 10 yr. The results of the tests made to date, as reported in previous reports to the committee, were summarized and discussed in a Symposium on Atmospheric Exposure Tests on Non-Ferrous Metals,[1] comprising six papers, held at this year's Spring Meeting of the Society in Pittsburgh.

The subcommittee, jointly with Committee B-7 on Light Metals and Alloys, is considering starting a new exposure test to consist principally of the light metals—aluminum and magnesium and their alloys. This program is in the preliminary planning stage at the present time.

Subcommittee VII on Weather (F. L. LaQue, chairman).—Following a policy of restricting activities during the war, Subcommittee VII was inactive up to the time of the meeting in Pittsburgh in February, 1946.

At that meeting it was decided to organize a subgroup to make a survey of methods and instruments currently available for measuring the weather factors thought to be important with respect to atmospheric corrosion. Whenever suitable methods and instruments are available, an effort will be made to correlate weather factors with the atmospheric corrosion of some common materials with the ultimate objective of improving the application of atmospheric corrosion data and the choice of sites for atmospheric corrosion tests.

It was also decided to reopen discussions with the Committee on Climatology of the American Geophysical Union in order to secure its cooperation in the work of this subcommittee.

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[1] Symposium on Atmospheric Exposure Tests on Non-Ferrous Metals (1946). (Symposium issued as separate publication.)

Subcommittee VIII on Galvanic and Electrolytic Corrosion (L. J. Gorman, chairman).—This subcommittee has a large program of work in progress.

TABLE I.—MATERIALS TO BE USED IN ASSEMBLING COUPLES FOR CORROSION TESTS.

PART I—DISK TYPE COUPLES.

(*A*) A.S.T.M. type AZ31X magnesium alloy to be coupled with:
1. Mild steel (0.05 to 0.08 Cu)
2. 85-15 brass, 1/2H
3. 24S-T aluminum alloy
4. 74S-T aluminum alloy
5. 56S aluminum alloy
6. 2S aluminum alloy
7. Stainless steel (type 304)
8. Monel metal
9. Nickel
10. Zinc
11. Cadmium-plated steel (0.0005 in.)
12. Zinc-plated steel (0.0005 in.)
13. A.S.T.M. Type M1 magnesium alloy

(*B*) A.S.T.M. type M1 magnesium alloy to be coupled with:
1. 85-15 brass, 1/2H
2. 56S aluminum alloy
3. 2S aluminum alloy
4. Zinc

PART II—BOLT TYPE COUPLES.
(0.030-in. Wire, Wound on 0.5-in. Threaded Bolts)

(*A*) AZ31X magnesium alloy wire to be wound on bolts of:
1. 85-15 brass
2. 2S aluminum
3. 36S aluminum
4. Zinc
5. M1 magnesium
6. AZ31X magnesium

(*B*) The following wires to be wound on bolts of AZ31X magnesium:
1. 85-15 brass wire
2. 2S aluminum wire
3. 56S aluminum wire
4. Zinc wire
5. M1 magnesium wire

PART III—PLATE TYPE COUPLES.
(Strips of Various Metals Bolted on Magnesium Plates with Various Bolt Materials)
AZ31X Magnesium Plates with the following bolts and strips:

Bolt Materials	Strip Materials					
	a	b	c	d	e	f
1. Mild steel	Mild steel	56S Al	Zinc	Cadmium	AZ31X Mg	M1Mg
2. 85-15 brass	85-15 brass	56S Al	Zinc	Cadmium	AZ31X Mg	M1Mg
3. 24S-T aluminum	24S-T aluminum	56S Al	Zinc	Cadmium	AZ31X Mg	M1Mg
4. 56S aluminum	56S aluminum	72S Al				
5. 61S-T aluminum	61S-T aluminum	56S Al				
6 Stainless steel	Stainless steel	56S Al	Zinc	Cadmium	AZ31X Mg	M1Mg
7. Monel metal	Monel metal	56S Al				
8. Nickel-plated brass	Nickel plated brass	56S Al				
9. Cadmium-plated steel	Cadmium-plated steel	56S Al				
10. Zinc-plated steel	Zinc-plated steel	56S Al				
11. Corronized steel	Corronized steel	56S Al				

Magnesium Tests.—The plans for the program of tests on the galvanic corrosion of magnesium alloys coupled with other metals and alloys have been completed, and the specimens and couples are being assembled. The tests will be made in three parts with three types of couples as follows:

Part I.—The disk type couple which is being used for the stainless steel tests now on exposure.

Part II.—The bolt and wire type couple developed by the Bell Telephone Laboratories.

Part III.—The plate and washer type couple, consisting of magnesium plates to which various bolt and washer materials are attached. The washer materials will be tested in the form of metal strips, bolted across the plates.

In Parts I and II, it is proposed to evaluate the amount of corrosion by inspection and weight changes. In Part III, it is planned to cut tension specimens from the magnesium plates and to obtain comparative tensile strength data on the corroded specimens and specimens cut from noncorroded plates.

The following test locations are to be used:

New York, N. Y.	Parts I, II and III
State College, Pa.	Parts I, II and III
Kure Beach, Wilmington, N. C.	Parts I, II and III
A tropical location	Parts I and III

Enough couples will be provided in Parts I and III for three exposure periods. The first period is to be 5 yr. and the duration of the second and third test periods will depend upon the results obtained in the first period. In Part II one set of couples will be exposed 1 yr., and three sets will be exposed in three consecutive periods of 4 months each during the same year.

The materials to be used in assembling couples in the various tests are given in Table I.

Stainless Steel Tests.—Disk samples of stainless steel coupled to other metals were exposed in 1941. One set will be removed this summer at the end of an exposure of 5 yr. The couples were exposed in New York, N. Y., State College, Pa., Altoona, Pa., and Kure Beach, N. C., and inspections made during the summer of 1945 indicated that in all cases the contact resistance of the couples was less than 0.1 ohm with the exception of the 24 ST aluminum couples at Kure Beach in which high resistance had developed.

This report has been submitted to letter ballot of the committee, which consists of 71 voting members; 45 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. S. RAWDON,
Chairman.

A. W. TRACY,
Secretary.

REPORT OF COMMITTEE B-4*

ON

ELECTRICAL-HEATING, ELECTRICAL-RESISTANCE AND ELECTRIC-FURNACE ALLOYS

Committee B-4 on Electrical-Heating, Electrical-Resistance, and Electric-Furnace Alloys held three meetings during the year: in Atlantic City, N. J., on June 13, 14, and 15, and in New York, N. Y., on November 14, 15, and 16, 1945, and on February 20, 21, and 22, 1946.

The committee consists of 67 members, of whom 28 are classified as producers, 32 as consumers, and 7 as general interest members.

The committee has approved revisions in its Regulations which provide for an Honorary Chairman (optional), Chairman, Vice-Chairman, and Secretary, and permits election of Honorary Members.

H. L. Curtis and Dean Harvey were elected honorary members.

Dean Harvey resigned as chairman of Committee B-4 and as representative of the Westinghouse Electric Corp. on March 31, 1946, on account of his retirement from the company. He has served as chairman since the organization of the committee in 1925. He was unanimously elected Honorary Chairman of the committee.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, J. W. Harsch.
Vice-Chairman, E. I. Shobert, II.
Secretary, F. E. Bash.

The 1945 Supplement to the Bibliography and Abstracts on Electrical Contacts, compiled by Subcommittee X on Contact Materials, was issued during the year. This second annual supplement contains 16 pages of references to articles on electrical contacts and circuit interruption, containing 59 references.

Subsequent to the 1945 Annual Meeting, Committee B-4 presented to the Society through the Administrative Committee on Standards a number of recommendations which are listed in Table I, together with the results of the letter ballot vote in Committee B-4 and the date of acceptance by the Administrative Committee. The new and revised tentatives have been given the designations indicated and appear in the 1946 Compilation of "A.S.T.M. Standards on Electrical-Heating, Electrical-Resistance, and Electric-Furnace Alloys."[1] This compilation sponsored by Committee B-4 contains 31 standards and has recently been published and should be of considerable value to industry.

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the Tentative Specifications for Drawn or Rolled Alloy, 80 per cent Nickel, 20 per cent Chromium, for Electrical Heating Elements (B 82 – 44 T) and Tentative Specifications for Drawn or Rolled Alloys, 60 per cent Nickel, 15 per cent Chromium, and Balance Iron, for Electrical Heating Elements (B 83 – 44 T)[2] be approved for reference to letter ballot of the Society for adoption as standard.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] Issued April, 1946.

TABLE I.–RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS, INCLUDING RECORD OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
New Tentative Specifications for:			
Nickel-Chromium-Iron Alloy Castings (35-15 Class) for High-Temperature Service (B 207 – 46 T), *accepted March 16, 1946*	43	0	8
New Tentative Methods of Test for:			
Strength of Welded Joints of Lead Wires for Electronic Devices and Lamps (B 203 – 45 T), *accepted January 16, 1946*	30	0	10
Surface Flaws in Tungsten Seal Rod and Wire (B 204 – 45 T), *accepted January 16, 1946*	26	0	14
Diameter by Weighing of Fine Wire Used in Electronic Devices and Lamps (B 205 – 45 T), *accepted January 16, 1946*	32	0	8
Revisions of Tentative Specifications for:			
Round Nickel Wire for Lamps and Electronic Devices (B 175 – 45 T), *accepted January 16, 1946*	29	0	11
Chromium-Nickel-Iron Alloy Castings (25-12 Class) for High-Temperature Service (B 190 – 45 T), *accepted January 16, 1946*	34	1	5
Revisions of Tentative Methods of Test for:			
Temper of Strip and Sheet Metals for Electronic Devices (B 155 – 45 T), *accepted January 16, 1946*	28	0	12
Testing Wire for Supports Used in Electronic Devices and Lamps (B 157 – 45 T), *accepted January 16, 1946*	33	0	7
Density of Fine Wire and Ribbon for Electronic Devices (B 180 – 45 T), *accepted January 16, 1946*	29	0	11
Life Test of Electrical Contact Materials (B 182 – 43 T), *accepted March 16, 1946*	43	0	8
Withdrawal of Standard Specifications for:			
Drawn or Rolled Alloy, 80 per cent Nickel, 20 per cent Chromium, for Electrical Heating Elements (B 82 – 41), *accepted March 16, 1946*	45	0	6
Drawn or Rolled Alloy, 60 per cent Nickel, 15 per cent Chromium, and Balance Iron, for Electrical Heating Elements (B 83 – 41), *accepted March 16, 1946*	45	0	6

This recommendation has been submitted to letter ballot of the committee which consists of 67 members; 51 members returned their ballots, with the following results: B 82, 45 affirmative, 0 negative and 6 "not voting"; B 83, 44 affirmative, 0 negative and 7 "not voting."

Activities of Subcommittees

Subcommittee II on Electrical Tests (P. H. Dike, chairman).—The subcommittee has reviewed all of the methods for making electrical tests on electrical-resistance and electrical-heating alloys, and has made minor revisions to bring them up to date.

Subcommittee V on Wrought and Cast Alloys for High-Temperature Use (J. W. Harsch, chairman).—The Tentative Specifications for Chromium-Nickel-Iron Alloy Castings (25-12 class) for High-Temperature Service (B 190 – 45 T) have been revised to include a brief appendix listing some of the more important technical papers on creep at elevated temperature.

New Tentative Specifications for Nickel-Chromium-Iron Alloy Castings (35-15 class) for High-Temperature Service (B 207 – 46 T) have been accepted by the Administrative Committee on Standards.

Consideration is being given to the preparation of specifications for alloy castings containing higher proportions of nickel and chromium.

Subcommittee VII on Thermostat Metals (P. H. Brace, chairman).—A study has been made of types of testing equipment to determine the construction most suitable for testing helical specimens of thermostat metal for equivalent yield stress in accordance with the Tentative Method of Test for Equivalent Yield Stress of Thermostat Metals (B 191 – 44 T) and a design is being prepared.

A method of test is under consideration for determining the comparative performance of mounted thermostat elements and a draft of the method is being prepared.

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[2] 1944 Book of A.S.T.M. Standards, Part I.

Microhardness test methods are being considered for determining the hardness of the components of thermostat metal. Hardness is of value as a control test.

Subcommittee VIII on Metallic Materials for Radio Tubes and Incandescent Lamps (S. A. Standing, chairman).—Three new tentative methods have been issued during the year and four existing tentatives have been revised. The titles of these appear in Table I.

The section on cathodes has been very active, collecting data and studying the effects of composition and other variables on the emissivity of oxide-coated cathodes. Five companies have reported on tests of a series of cathode melts in diodes. Chemical and metallurgical studies are being made on the material. Experimental work is continuing.

A study is being made on the methods of tension and elongation testing of fine wire.

A study is also being made of the magnetic permeability of slightly magnetic materials used in lamps and electronic devices.

Continued studies are being made of particle size and density of powders. Arrangements have been completed for interlaboratory testing by various methods.

A draft of a specification for glass sealing alloys has been prepared and is being circulated in the industry.

Subcommittee IX on Methods of Test for Alloys in Controlled Atmospheres (P. H. Brace, chairman).—It is planned to resume the corrosion tests in controlled atmospheres as used in electric furnaces at elevated temperature. These tests were postponed during the war. Tests are to be made to determine the effect of humidity in hydrogen atmosphere and of sulfur and humidity in "combusted gas."

Subcommittee X on Contact Materials (F. E. Carter, chairman).—Tentative Method for Life Test of Electrical Contact Materials (B 182 – 46 T) has been revised to specify the minor changes which were found necessary in the testing machine in order to obtain reproducible results with different machines. Tests have been made by four companies with the improved machines to determine the no-weld current and loss of weight of silver and nickel contact material. These tests showed good agreement.

Arrangements have been made for a study in seven laboratories of the following five tests of the electrical characteristics of contacts: (1) surety of making a circuit, (2) welding characteristic, (3) arcing characteristic, (4) contact resistance build-up, and (5) wear. These characteristics will be determined for fine silver, tungsten, nickel, cadmium, copper, and coin silver.

A Tentative Method of Test for Hardness of Contact Materials is being prepared, including the superficial Rockwell test and the Vickers test.

E. I. Shobert II presented before the subcommittee a paper on "Welding or Sticking of Electrical Contacts." He pointed out that there was good agreement between the no-weld currents of contact materials determined by test and those calculated theoretically from the physical properties of these materials. This paper appears in the compilation of B-4 standards[1] and also has been published separately.

Proposed standards for dimensions of composite contact-tipped screws and studs consisting of silver and silver-base contacts, and of tungsten and molybdenum refractory composition tips for use on composite rivet-type contacts have been prepared. These are now under consideration in Section C on

Standardization of Contact Forms and Sizes (A. B. Gibson, chairman), in cooperation with the National Electrical Manufacturers Association.

This report has been submitted to letter ballot of the committee, which consists of 67 members; 51 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. W. HARSCH,
Acting Chairman.

F. E. BASH,
Secretary.

REPORT OF COMMITTEE B-5*

ON

COPPER AND COPPER ALLOYS, CAST AND WROUGHT

Committee B-5 on Copper and Copper Alloys, Cast and Wrought, held two meetings during the year at New York, N. Y., on September 12, 1945, and March 12, 1946. At these sessions the Advisory Committee and Subcommittees A-1, A-2, A-3, A-4, B-1, and C-1 also met.

During the year 21 members were added to the committee and there were seven removals. At the present time the committee consists of 134 members, of whom 114 are voting members; 49 are classified as producers, 48 as consumers, and 17 as general interest members.

Subsequent to the 1946 Annual Meeting, Committee B-5 presented to the Society through the Administrative Committee on Standards a number of recommendations which are listed in Table I, together with the results of the letter ballot vote in Committee B-5 and the date of acceptance by the Standards Committee. The new and revised tentatives have been given the designations indicated and together with the tentative revisions appear in the April, 1946 Compilation of "A.S.T.M. Standards on Copper and Copper Alloys."

At the meeting on March 12, 1946, the election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, G. H. Harnden.

First Vice-Chairman, R. A. Wilkins.

Second Vice-Chairman, J. J. Kanter.

Secretary, V. P. Weaver.

TABLE I. RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS ON JANUARY 4, 1946, INCLUDING RECORD OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
New Tentative Specifications for:			
Copper-Nickel-Zinc Alloy Wire (B 206 – 46 T)	34	1	30
Revision of Tentative Specifications for:			
Naval Brass Rods, Bars and Shapes (B 21 – 46 T)	28	1	36
Seamless Copper Tubes (B 75 – 46 T)	27	1	37
Copper-Nickel-Zinc and Copper-Nickel Alloy Sheet and Strip (B 122 – 46 T)	21	1	43
Copper Rods, Bars and Shapes (B 133 – 46 T)	28	1	36
Miscellaneous Brass Tubes (B 135 – 46 T)	27	1	37
Phosphor Bronze Rods, Bars and Shapes (B 139 – 46 T)	28	1	36
Aluminum Bronze Rods, Bars and Shapes (B 150 – 46 T)	28	1	36
Copper-Nickel-Zinc Alloy Rod and Bar (B 151 – 46 T)	28	1	36
Phosphor Bronze Wire (B 159 – 46 T)	30	1	34
Beryllium-Copper Alloy Strip (B 194 – 46 T)	20	1	45
Beryllium-Copper Alloy Rod and Bar (B 196 – 46 T)	28	1	36
Beryllium-Copper Alloy Wire (B 197 – 46 T)	30	1	34
Tentative Revision of Standard Specifications for:			
Copper Rods for Locomotive Staybolts (B 12 – 45)	28	1	36
Free-Cutting Brass Rod and Bar for Use in Screw Machines (B 16 – 45)	28	1	36
Copper Water Tube (B 88 – 45)	24	1	40
Copper-Silicon Alloy Rods, Bars and Shapes (B 98 – 45)	28	1	36
Copper-Silicon Alloy Wire for General Purposes (B 99 – 45)	30	1	34
Copper and Copper-Alloy Seamless Condenser Tubes and Furrule Stock (B 111 – 45)	27	1	37
Brass Wire (B 134 – 45)	30	1	34
Manganese Bronze Rods, Bars and Shapes (B 138 – 45)	28	1	36
Leaded Red Brass (Hardware Bronze) Rods, Bars and Shapes (B 140 – 45)	28	1	36

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

TABLE II.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE			
Rec. Practice for Preparing Tension Test Specimens for Copper-Base Alloy Castings	45	0	27
II. REVISIONS OF TENTATIVE SPECIFICATIONS			
Spec. for Cartridge Brass Sheet, Strip, Plate, Bar, and Disks (B 19 - 44 T)	43	1	28
Spec. for Naval Brass Rods, Bars, and Shapes (B 21 - 46 T)	43	0	29
Spec. for Bronze Castings for Turntables and Movable Bridges and for Bearing and Expansion Plates of Fixed Bridges (B 22 - 45 T)	44	0	28
Spec. for Brass Sheet and Strip (B 36 - 44 T)	42	1	29
Spec. for Seamless Copper Tubes (B 75 - 46 T)	43	1	28
Spec. for Leaded Brass Sheet and Strip (B 121 - 44 T)	40	1	31
Spec. for Copper-Nickel-Zinc and Copper-Nickel Alloy Sheet and Strip (B 122 - 46 T)	42	0	30
Spec. for Cartridge Brass Cartridge Case Cups (B 129 - 44 T)	37	1	34
Spec. for Gilding Metal Strip (B 130 - 44 T)	38	1	33
Spec. for Gilding Metal Bullet Jacket Cups (B 131 - 44 T)	36	0	36
Spec. for Leaded High-Strength Yellow Brass (Manganese Bronze) Sand Castings (B 132 - 44 T)	43	0	29
Spec. for Copper Rods, Bars, and Shapes (B 133 - 46 T)	44	0	28
Spec. for Miscellaneous Brass Tubes (B 135 - 46 T)	41	1	30
Spec. for Phosphor Bronze Rods, Bars, and Shapes (B 139 - 46 T)	44	0	28
Spec. for Tin-Bronze and Leaded Tin-Bronze Sand Castings (B 143 - 44 T)	46	0	26
Spec. for High-Leaded Tin-Bronze Sand Castings (B 144 - 45 T)	47	0	25
Spec. for Leaded Red Brass and Leaded Semi-Red Brass Sand Castings (B 145 - 45 T)	46	0	26
Spec. for Leaded Yellow Brass Sand Castings for General Purposes (B 146 - 45 T)	46	0	26
Spec. for High-Strength Yellow Brass (Manganese Bronze) and High-Strength Leaded Yellow Brass (Leaded Manganese Bronze) Sand Castings (B 147 - 44 T)	45	0	27
Spec. for Aluminum-Bronze Sand Castings (B 148 - 45 T)	42	0	30
Spec. for Leaded Nickel-Brass (Leaded Nickel-Silver) and Leaded Nickel-Bronze (Leaded Nickel-Silver) Sand Castings (B 149 - 44 T)	43	0	29
Spec. for Aluminum Bronze Rods, Bars, and Shapes (B 150 - 46 T)	43	0	29
Spec. for Copper-Nickel-Zinc Alloy Rod, Bar, and Wire (B 151 - 46 T)	42	0	30
Spec. for Copper Sheet, Strip, and Plate (B 152 - 45 T)	43	1	28

TABLE II—*Continued*

Items	Affirmative	Negative	Ballots Marked "Not Voting"
II. REVISIONS OF TENTATIVE SPECIFICATIONS—*Continued*			
Spec. for Phosphor Bronze Wire (B 159 - 46 T)	41	0	31
Spec. for Aluminum Bronze Sheet and Strip (B 169 - 44 T)	38	1	33
Spec. for Copper Bus Bars, Rods, and Shapes (B 187 - 45 T)	39	1	32
Spec. for Copper Bus Pipes and Tubes (B 188 - 45 T)	39	1	32
Spec. for Beryllium-Copper Alloy Strip (B 194 - 46 T)	39	0	33
Spec. for Beryllium-Copper Alloy Strip, Special Grade (B 195 - 45 T)	37	1	34
Spec. for Beryllium-Copper Alloy Rod and Bar (B 196 - 46 T)	38	0	34
Spec. for Beryllium-Copper Alloy Wire (B 197 - 46 T)	37	0	35
Spec. for Silicon-Bronze and Silicon-Brass Sand Castings (B 198 - 45 T)	44	0	28
Spec. for Copper-Nickel-Zinc Alloy Wire (B 206 - 46 T)	39	0	33
III. REVISION OF STANDARDS, IMMEDIATE ADOPTION			
Spec. for Copper Plates for Locomotive Fireboxes (B 11 - 41)	36	1	35
Spec. for Copper Bars for Locomotive Staybolts (B 12 - 45)	36	0	36
Spec. for Free-Cutting Brass Rod and Bar for Use in Screw Machines (B 16 - 45)	45	0	27
Spec. for Copper Pipe, Standard Sizes (B 42 - 45)	42	2	28
Spec. for Brass Pipe, Standard Sizes (B 43 - 45)	41	2	29
Spec. for Phosphor Copper (B 52 - 43)	41	0	31
Spec. for Silicon Copper (B 53 - 43)	40	0	32
Spec. for Steam or Valve Bronze Castings (B 61 - 44)	44	0	28
Spec. for Composition Brass or Ounce Metal Castings (B 62 - 44)	45	0	27
Spec. for Bronze Castings in the Rough for Locomotive Wearing Parts (B 66 - 44)	36	0	36
Spec. for Car and Tender Journal Bearings, Lined (B 67 - 44)	33	0	39
Spec. for Seamless Copper Tubing, Bright Annealed (B 68 - 45)	44	1	27
Spec. for Copper Water Tube (B 88 - 45)	41	1	30
Spec. for Copper-Silicon Alloy Plate and Sheet for Pressure Vessels (B 96 - 42)	41	1	30
Spec. for Copper-Silicon Alloy Sheet and Strip for General Purposes (B 97 - 44)	41	1	30
Spec. for Copper-Silicon Alloy Rods, Bars, and Shapes (B 98 - 45)	42	0	30
Spec. for Copper-Silicon Alloy Wire for General Purposes (B 99 - 45)	41	0	31
Spec. for Rolled Copper-Alloy Bearing and Expansion Plates for Bridge and Other Structural Uses (B 100 - 44)	38	1	33
Spec. for Phosphor Bronze Sheet and Strip (B 103 - 44)	41	1	30
Spec. for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 - 45)	44	0	28

TABLE II—*Concluded*

Items	Affirmative	Negative	Ballots Marked "Not Voting"
III. REVISION OF STANDARDS, IMMEDIATE ADOPTION—*Cont'd*			
Spec. for Copper-Base Alloy Forging Rods, Bars, and Shapes (B 124 - 45)	41	1	30
Spec. for Brass Wire (B 134 - 45)	41	0	31
Spec. for Manganese Bronze Rods, Bars, and Shapes (B 138 - 45)	43	0	29
Spec. for Leaded Red Brass (Hardware Bronze) Rods, Bars, and Shapes (B 140 - 45)	41	0	31
Spec. for Copper-Alloy Condenser Tube Plates (B 171 - 45)	40	1	31
IV. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS			
Spec. for Copper Bars for Locomotive Staybolts (B 12 - 45)	37	1	34
Spec. for Free-Cutting Brass Rod and Bar for Use in Screw Machines (B 16 - 45)	46	0	26
Spec. for Copper Water Tube (B 88 - 45)	42	0	30
Spec. for Copper-Silicon Alloy Rods, Bars, and Shapes (B 98 - 45)	42	0	30
Spec. for Copper-Silicon Alloy Wire for General Purposes (B 99 - 45)	41	0	31
Spec. for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 - 45)	45	0	27
Spec. for Brass Wire (B 134 - 45)	41	0	31
Spec. for Manganese Bronze Rods, Bars, and Shapes (B 138 - 45)	44	0	28
Spec. for Leaded Red Brass (Hardware Bronze) Rods, Bars, and Shapes (B 140 - 45)	42	0	29

WITHDRAWAL OF EMERGENCY ALTERNATE PROVISIONS

Certain of the revisions being recommended in the standard and tentative specifications include changes now published as emergency alternate provisions. For the most part, these include provisions for test bars for castings; in the remainder the emergency provisions are no longer necessary. The committee accordingly recommends that, concurrently with the approval of the recommendations affecting standards in this report, the following emergency alternate provisions (all now remaining under the jurisdiction of the committee) be withdrawn:

Emergency Alternate Provisions in Specifications for:

Free-Cutting Brass Rod and Bar for Use in Screw Machines (EA - B 16),
Bronze Castings for Turntables and Movable Bridges and for Bearing and Expansion Plates of Fixed Bridges (EA - B 22b),
Steam or Valve Bronze Castings (EA - B 61),
Composition Brass or Ounce Metal Castings (EA - B 62b),
Tin-Bronze and Leaded Tin-Bronze Sand Castings (EA - B 143b),
High-Leaded Tin-Bronze Sand Castings (EA - B 144b),
Leaded Red Brass and Leaded Semi-Red Brass Sand Castings (EA - B 145b), and
Leaded Yellow Brass Sand Castings for General Purposes (EA - B 146b)

RECOMMENDATIONS AFFECTING STANDARDS

The committee is submitting one new tentative recommended practice, revisions in 34 tentative specifications, revisions in 25 standards for immediate adoption, and the adoption of 9 tentative revisions as standard. The standards and tentatives affected are listed in Table II and the revisions recommended are given in detail in the Appendix.

The recommendations in this report have been submitted to letter ballot of the committee, which consists of 114 voting members, with the results shown in Table II.

ACTIVITIES OF THE ADVISORY COMMITTEE

On the recommendation of the Advisory Committee, Committee B-5 approved the following resolution:

RESOLUTION ON THE DEATH OF C. H. DAVIS

Committee B-5 of the American Society for Testing Materials records with sorrow the death of Charles Hollister Davis on May 22, 1945.

In his passing, the Society has lost a distinguished and respected collaborator and a sagacious and aggressive member who made substantial contributions to the work of the committee and to the welfare of the Society. As secretary of the committee for many years, he was instrumental in formulating its policies and guiding its deliberations.

As metallurgist and administrator, he showed outstanding executive ability and a thorough understanding of technical problems. His work was based on a sound scientific background enriched by long experience in research where his achievements won the merited esteem of his associates.

He was open minded, patient and tolerant of the opinions of others, and the memory of his example will inspire his associates to carry on in his resolute spirit. By kindly personal contact he endeared himself to each member of the committee and each member has experienced the loss of a true friend.

The following revisions of the regulations of the committee were approved by letter ballot:

Article IV, Section 6.—Renumber as Section 7 and add a new Section 6 to read as follows: "Sec. 6. Vacancies in elective office shall be filled by appointment by the Advisory Committee. Such an appointee shall serve until the next regular biennial election of officers."

Article VI, Section 2.—Add a new sentence to the last paragraph of this section to read as follows: "Officers of subcommittees shall serve until their successors have been appointed."

Article VII, Section 1.—Change the first sentence to read as follows: "Proposed standards or proposed revisions of existing standards shall be referred to a letter ballot vote of the originating subcommittee and shall receive the approval of at least nine-tenths of those voting "Yes" or "No" for submission to the main committee. Providing further that if less than two-thirds of the subcommittee are recorded as voting "Yes," "No," or "Not Voting" reference to letter ballot of the main committee shall be subject to Advisory Committee approval."

ACTIVITIES OF SUBCOMMITTEES

The following is the report of the several subcommittees during the year. The actions recommended at the Fall Meeting were approved by the Administrative Committee on Standards on January 4, 1946, and are listed in detail earlier in this report. The actions at the Spring Meeting are given in detail in the Appendix. Included in the recommendations are the cancellations of all Emergency Alternate Provisions of the committee's specifications.

Subcommittee A-1 on Plate, Sheet, and Strip (R. A. Frye, chairman, R. A. Lubker, secretary) recommended at the Fall Meeting that revisions be incorporated in tentative Specifications B 122 and B 194. A task group (G. R. Gohn, chairman) prepared the revisions for Specifications B 122.

At the Spring Meeting the subcommittee recommended that revisions be incorporated in 11 tentative specifications: B 19, B 36, B 121, B 122, B 129, B 130, B 131, B 152, B 169, B 194, and B 195. Revisions for immediate adoption were recommended in six standard specifications: B 11, B 96, B 97, B 100, B 103, and B 171. These recommendations, it will be noted, include adding in the appendix of the eight general specifications for sheet and strip a table of preferred thicknesses based on the American Standard B32.1.

A task group (N. H. Murdza, chairman) prepared the revisions for specifications B 129 and B 131.

Among the subjects being studied by the subcommittee is the question of the inclusion of requirements for plate and rolled bar in the sheet and strip specifications, and a review of the grain size requirements in the Specifications for Sheet Brass (B 36).

Subcommittee A-2 on Rods, Bars, and Shapes (H. C. Ashley, chairman) recommended that Emergency Alternate Provisions EA-B 16 applying to the Standard Specifications for Free-Cutting Brass Rod and Bar for Use in Screw Machines (B 16 – 45) be withdrawn.

At the Fall Meeting it was recommended that revisions be incorporated in six tentative specifications: B 21, B 133, B 139, B 150, B 151, and B 196. Tentative revisions were recommended in five standard specifications: B 12, B 16, B 98, B 138, and B 140.

A task group (B. J. Sirois, chairman) prepared the definitions of edge contours incorporated in the specifications. Incorporation of dimensional tolerances for piston finish and shafting rod in Specifications B 21, B 138, B 139, and B 150 was at the recommendation of Subcommittee C-2.

At the Spring Meeting the subcommittee recommended that revisions be incorporated in seven tentative specifications: B 21, B 133, B 139, B 150, B 151, B 187, and B 196. Revisions for immediate adoption were recommended in Standard Specifications B 124. Revisions for immediate adoption and the adoption of tentative revisions were recommended in five standard specifications: B 12, B 16, B 98, B 138 and B 140.

A task group (G. Williams, Jr., chairman) prepared the revisions of chemical limits for Specifications B 150.

Revision in the straightness tolerances for Specifications B 187 was made at the recommendation of Subcommittee C-2.

Among the subjects under review in the subcommittee are the preparation of new specifications for copper-nickel-zinc rod and bar to replace Specifications B 151 and the possible revision of the tempers and physical properties in the Specifications for Free-Cutting Brass Rod (B 16).

Subcommittee A-3 on Wire and Wire Rod (B. J. Sirois, chairman) recommended at the Fall Meeting new Tentative Specifications for Copper-Nickel-Zinc Alloy Wire (B 206) and revisions in Tentative Specifications B 159 and B 197. It also recommended tentative revisions in Standard Specifications B 99 and B 134.

A task group (B. J. Sirois, chairman) prepared the standard edge and corner radii for incorporation in the specifications.

At the Spring Meeting the subcommittee recommended that revisions be incorporated in three tentative specifications: B 159, B 197 and B 206. Revisions for immediate adoption and the adoption of tentative revisions were recommended in Standard Specifications B 99 and B 134.

Among the subjects currently being studied in the subcommittee are the possibility of substituting maximum tensile strength values instead of minimum elongation in the Specifications for Copper-Silicon Alloy Wire (B 99) and Beryllium-Copper Alloy Wire (B 197). Another subject under discussion is the addition of a new alloy and the revision of the physical properties in the Specifications for Copper-Nickel-Zinc Alloy Wire (B 206).

Subcommittee A-4 on Pipe and Tube (Alan Morris, chairman, R. S. Pratt, secretary) recommended at the Fall Meeting that revisions be incorporated in Tentative Specifications B 75 and B 135. Tentative revisions were recommended in Standard Specifications B 88 and B 111.

A task group (H. C. Ashley, chairman) prepared the revisions of physical properties for Specifications B 75 and B 135.

At the Spring Meeting it was recommended that revisions be incorporated in three tentative specifications: B 75, B 135, and B 188. Revisions for immediate adoption were recommended in three standard specifications: B 42, B 43, and B 68. Revisions for immediate adoption and the adoption of tentative revisions were recommended in Standard Specifications B 88 and B 111.

A task group (G. R. Gohn, chairman) prepared the flattening test for incorporation in six Specifications: B 42, B 43, B 68, B 75, B 88, and B 135.

This subcommittee is reviewing the

tensile requirements in the Specifications for Copper Water Tube (B 88) to bring them in line with those in the Specifications for Seamless Copper Tubes (B 75). Another subject being studied is the chemical limits in the various tube specifications to bring them in line with the chemical requirements for similar alloys in other products.

Subcommittee B-1 on Castings and Ingots for Remelting (G. H. Clamer, chairman, C. S. Cole, secretary) recommended that the Emergency Alternate Provisions EA-B 22b, EA-B 61, EA-B 62b, EA-B 143b, EA-B 145b, and EA-B 146b be withdrawn concurrently with the approval of the new proposed Tentative Recommended Practice for Preparing Tension Test Specimens for Copper-Base Alloy Castings.[1] At the same time the sections for the test bar coupons and test specimens appearing in the various specifications were recommended to be deleted and replaced by appropriate reference to this new recommended practice. This involved changes in Standard Specifications B 61 and B 62, and in 10 tentative specifications: B 22, B 132, B 143, B 144, B 145, B 146, B 147, B 148, B 149, and B 198.

The subcommittee voted to set up a task group to study the subject of test bars and test bar practice which will review the work currently being done on this subject at Battelle Memorial Institute under the sponsorship of the Non-Ferrous Ingot Metal Institute;[2] the work on the same subject at the Naval Research Laboratory and the British Standards Institution Code of Procedure in Inspection of Copper-Base Alloy Castings which was recently published in England. The new recommended practice will later be expanded as the task group has recommendations to offer.

Revisions for immediate adoption were recommended in four standard specifications: B 52, B 53, B 66, and B 67.

Subcommittee C-1 on Methods of Test (G. R. Gohn, chairman) recommended the adoption of a standard rejection clause prepared by a task group (C. J. Snyder, chairman) for incorporation in the specifications under the jurisdiction of Subcommittees A-1, A-2, A-3, and A-4.

A task group (J. F. Ednie, chairman) reviewed the significance of numerical limitations for chemical composition and physical properties and the subcommittee recommended that the findings of the group be referred to Subcommittees A-1, A-2, A-3, A-4, and B-1 for application to the various specifications under their jurisdiction.

The representative of Committee B-5 on Committee E-1 on Methods of Testing obtained recommendations from the Section on Indentation Hardness relating to the location of Rockwell and Brinell tests on sheet and rod stock.

Future work of the subcommittee includes the development of methods of tension testing for thin sheet metals and for rod stock.

Subcommittee C-2 on Tolerances (J. E. McGraw, chairman) recommended to Subcommittee A-2 the incorporation of dimensional tolerances for piston rod and shafting in Specifications B 21, B 138, B 139, and B 150 and revision in the straightness tolerances for Specifications B 187.

At the request of Subcommittees A-1, A-2, A-3, A-4, and C-1, the subcommittee obtained from the Copper & Brass Research Ass'n. a statement as to the significance of values for dimensional tolerances and recommended its adoption in the applicable specifications under Committee B-5.

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[1] This recommended practice was accepted as tentative by the Society and appears in the 1946 Book of A.S.T.M. Standards, Part I B.

[2] This work was reported by Mr. Clamer in his lecture "Test Bars for 85-5-5-5 Alloy, Their Design and Some Factors Affecting Their Properties," which was presented at the 1946 American Foundrymen's Association Golden Anniversary Convention May 9, 1946, Cleveland, Ohio.

Recommendations as to the maximum thickness limitation of width tolerances for slit metal were presented to Subcommittee A-1 for adoption in the applicable specifications. The subcommittee recommended to Subcommittee A-4 revisions in the out-of-roundness tolerances in Specifications B 75 and B 135.

The subcommittee is currently discussing with CABRA the latter's proposed new classification of flat products which involves terminology differing from that now used in A.S.T.M. specifications.

Subcommittee C-3 on Editorial and Publications (W. F. Roeser, chairman) has been assigned the development of definitions of terms relating to copper and brass, the development of a classification of wrought copper alloys, and the coordination of phraseology in Committee B-5 specifications.

This report has been submitted to letter ballot of the committee, which consists of 114 voting members; 72 members returned their ballots, of whom 61 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

G. H. HARNDEN,
Chairman.

V. P. WEAVER,
Secretary.

EDITORIAL NOTE

The emergency alternate provisions listed in the report were discontinued by action of the Administrative Committee on Standards on September 9, 1946.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR COPPER AND COPPER ALLOYS, CAST AND WROUGHT

In this Appendix are given the recommendations affecting certain standards covering copper and copper alloys which are referred to earlier in this report. The standards appear in their present form in the 1944 Book of A.S.T.M. Standards, Part I, or the 1945 Supplement to Book of A.S.T.M. Standards, Part I, and the compilation of "A.S.T.M. Standards on Copper and Copper Alloys," April, 1946.

Revisions of Tentative Specifications

The committee recommends that the following 34 tentative specifications be revised as indicated below and continued as tentative.

Tentative Specifications for Cartridge Brass Sheet, Strip, Plate, Bar, and Disks (B 19 – 44 T):

Section 4.—Delete the last zero in each of the limits for chemical composition; also delete Note 2.

Section 15.—In the table for slit metal, column 1, change "0.182" to "0.187."

Sections 14, 15, 16, 17, and 18.—Add a note to read as follows: "Note.—The dimensional tolerance requirements of these specifications are absolute. Measurements to determine compliance with the specified tolerances should be made with a *practical* instrument having divisions at least as fine as the degree of precision of the limiting dimension, that is, the nominal dimension plus or minus the tolerance."

Section 23.—Revise to read as follows: "Material that fails to conform to these specifications will be rejected, and the manufacturer shall be notified within 30 days after receipt of the material."

Tentative Specifications for Naval Brass Rods, Bars, and Shapes (B 21 – 46 T):

Section 2.—Revise Item 3 to read as follows: "Diameter or distance between parallel surfaces: if tolerances other than plus and minus are required, whether all plus or all minus, (Section 12)."

Section 7.—Revise to read as follows: "For grade A material, the bend test specimen shall stand being bent cold through the angle prescribed in Table II around a pin, the diameter of which is equal to twice the diameter or thickness of the specimen, without fracturing."

Section 9.—In Paragraph (*a*), line 15, change the word "rods" to "material." In Paragraphs (*b*) and (*c*), line 2, change the word "rod" to "material."

Section 10.—In Paragraph (*a*), insert the words "bars or shapes" after "rods." In Paragraph (*b*), line 3, change "rod" to "length."

Sections 12, 13, 14, and 16.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Table VII.—Change the title of Table VII to read as follows: "Straightness Tolerances. Applicable to Any Longitudinal Surface or Edge."

Section 17.—Revise to read as follows: "The material shall be free from injurious defects, and shall have a bright, smooth surface."

Tentative Specifications for Bronze Castings for Turntables and Movable Bridges and for Bearing and Expansion Plates of Fixed Bridges (B 22 – 45 T):

Section 8.—Revise this section to read as follows:

> 8. (*a*) It is recommended that the tension test specimens and coupons for tension tests be prepared in accordance with the Tentative Recommended Practice for Preparing Tension Test Specimens for Copper-Base Alloy Castings (A.S.T.M. Designation: B 208),[1] except that Fig. 2 of B 208 is not applicable to these alloys.
>
> (*b*) A suitable test bar for the compression test specimen shall be an integral part of the casting and shall be fed and cooled under the same conditions as the castings.
>
> (*c*) Compression test specimens shall be cylinders 1 sq. in. in cross-sectional area and 1 in. in height.[2]

Figures 1 and 2.—Delete the present Figs. 1 and 2.

Tentative Specifications for Brass Sheet and Strip (B 36 – 44 T):

Section 1.—Delete alloys Nos. 5 and 7 from the table appearing in Paragraph (*a*), and add a footnote to read as follows: "The former alloys Nos. 5 and 7 have been discontinued."

Section 4.—Delete alloys Nos. 5 and 7 from the table appearing in this section and add a footnote to read as follows: "The former alloys Nos. 5 and 7 have been discontinued." For alloy No. 8 change maximum copper "67.5" to "68.0" and change lead, maximum, "0.30" to "0.15."

Section 7 (b).—Delete all reference to alloys Nos. 5 and 7.

Tables I, II, and III.—Delete all reference to alloys Nos. 5 and 7.

Table V.—In the third column of the table for slit metal change "0.182" to "0.187."

Sections 14, 15, 16, 17, and 18.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Section 21.—Add the words "within 30 days after receipt of the material."

New Appendix.—Add an appendix to read as follows: "It is recommended that wherever possible material purchased to these specifications be ordered in thicknesses listed as follows:

PREFERRED THICKNESSES FOR UNCOATED THIN FLAT-ROLLED METALS[a] (UNDER 0.250 IN.) (ALL VALUES IN INCHES).

	0.006	0.012	0.025	0.050	0.100	0.200
	0.007	0.014	0.028	0.056	0.112	0.224
0.004	0.008	0.016	0.032	0.063	0.125	
	0.009	0.018	0.036	0.071	0.140	
0.005	0.010	0.020	0.040	0.080	0.160	
	0.011	0.022	0.045	0.090	0.180	

[a] In conformance with American Standard B 32.1.

Tentative Specifications for Seamless Copper Tubes (B 75 – 46 T):

Section 8.—Reletter as Paragraph (*a*) and add the following as Paragraph (*b*): "As an alternate to the expansion test for tubes over 4 in. in diameter in the annealed condition, a section 4 in. in length shall be cut from the end of one of the lengths for a flattening test. This 4-in. sample shall be flattened so that a gage set at three times the wall thickness will pass over the tube freely throughout the flattened part. The tubes so tested shall develop no cracks or flaws visible to the unaided eye as a result of this test. In making the flattening test the elements shall be slowly flattened by one stroke of the press."

Sections 16 and 17.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Table II.—In Table II, delete the column headed "Stubs' Gage."

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[1] This recommended practice was accepted as tentative by the Society and appears in the 1946 Book of A.S.T.M. Standards, Part I-B.

[2] The matters of revising the dimensions of compression test specimens and the method of determining the so-called "deformation limit" are under consideration by Committee E-1 on Methods of Testing. Tests are being made on cylindrical specimens 1 sq. in. in cross-sectional area and 3 in. in height. Committee B-5 on Copper and Copper Alloys, Cast and Wrought, which has written these specifications feels that these specifications with the present compression tests as specified may be satisfactorily used until better compression test specimens are available.—ED.

Tentative Specifications for Leaded Brass Sheet and Strip (B 121 – 44 T):

Table V.—In the third column of the table for slit metal change "0.182" to "0.187."

Sections 14, 15, 16, 17, and 18.—Add a note as indicated above for Sections 14 to 18 of Specifications B 19.

Section 21.—Add the words "within 30 days after receipt of the material."

New Appendix.—Add an appendix as indicated above for Specifications B 36.

Tentative Specifications for Copper-Nickel-Zinc and Copper-Nickel Alloy Sheet and Strip (B 122 – 46 T):

Sections 14, 15, 16, and 17.—Add a note as indicated above for Sections 14 to 18 of Specifications B 19.

New Appendix.—Add an appendix as indicated above for Specifications B 36.

Tentative Specifications for Cartridge Brass Cartridge Case Cups (B 129 – 44 T):

The proposed revision of these specifications includes changes in manufacturing and chemical requirements, deletion of all requirements for sheet and strip, changes in the grain size requirements, addition of a section on retests and revision of the rejection clause. The revised specifications are appended hereto.[3]

Tentative Specifications for Gilding Metal Strip (B 130 – 44 T):

Section 4.—Delete Note 2 and revise the limits of chemical composition to read as follows:

Copper, per cent	89.0 to 92.0
Lead, max., per cent	0.05
Iron, max., per cent	0.05
Total elements, other than copper and zinc, max., per cent	0.13
Zinc	remainder

Sections 14, 15, 16, and 17.—Add a note as indicated above for Sections 14 to 18 of Specifications B 19.

Section 22.—Add the words "within 30 days after receipt of the material."

Tentative Specifications for Gilding Metal Bullet Jacket Cups (B 131 – 44 T):

The proposed revision of these specifications includes changes in anneals, manufacturing and chemical requirements, deletion of all requirements on metal for cups, changes in grain size, addition of a section on retests and revision of the rejection clause. The revised specifications are appended hereto.[3]

Tentative Specifications for Leaded High-Strength Yellow Brass (Manganese Bronze) Sand Castings (B 132 – 44 T):

Section 5.—Revise Section 5 to read as follows: "It is recommended that the tension test specimens and coupons for tension tests be prepared in accordance with the Tentative Recommended Practice for Preparing Tension Test Specimens for Copper-Base Alloy Castings (A.S.T.M. Designation: B 208),[1] except that Figs. 1, 3 and 4 of B 208 are not applicable to these alloys."

Figures 1, and 2.—Delete the present Figs. 1 and 2.

Tentative Specifications for Copper Rods, Bars, and Shapes (B 133 – 46 T):

Sections 18, 19, 20, and 21.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Table VI.—Change the title of Table VI to read as follows: "Straightness Tolerances. Applicable to Any Longitudinal Surface or Edge."

Tentative Specifications for Miscellaneous Brass Tubes (B 135 – 46 T):

Section 10.—Add a new Paragraph (*b*) to read as indicated above for Section

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[3] These revised specifications were accepted by the Society and appear in the 1946 Book of A.S.T.M. Standards, Part I-B.

8 (*b*) in Specifications B 75. Reletter the present Paragraph (*b*) as Paragraph (*c*) and change the last two words to read "these tests."

Sections 15 and 16.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Table III.—In Table III delete the column headed "Stubs' Gage."

Tentative Specifications for Phosphor Bronze Rods, Bars, and Shapes (B 139 – 46 T):

Sections 12, 13, 14, and 16.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Table VII.—Change the title of Table VII to read as follows: "Straightness Tolerances. Applicable to Any Longitudinal Surface or Edge."

Section 17.—Change to read as follows: "The material shall be free from injurious defects, and unless otherwise specified shall have a bright, smooth surface."

Tentative Specifications for Tin-Bronze and Leaded Tin-Bronze Sand Castings (B 143 – 44 T),

Tentative Specifications for High-Leaded Tin-Bronze Sand Castings (B 144 – 45 T),

Tentative Specifications for Leaded Red Brass and Leaded Semi-Red Brass Sand Castings (B 145 – 45 T), and

Tentative Specifications for Leaded Yellow Brass Sand Castings for General Purposes (B 146 – 45 T):

Section 9.—Revise to read as follows: "9. It is recommended that the tension test specimens and coupons for tension tests be prepared in accordance with the Tentative Recommended Practice for Preparing Tension Test Specimens for Copper-Base Alloy Castings (A.S.T.M. Designation: B 208),[1] except that Fig. 2 of B 208 is not applicable to these alloys."

Figures 1 and 2.—Delete the present Figs. 1 and 2.

Tentative Specifications for High-Strength Yellow Brass (Manganese Bronze) and High-Strength Leaded Yellow Brass (Leaded Manganese Bronze) Sand Castings (B 147 – 44 T):

Section 8.—Revise to read as follows: "8. Unless special authority to use coupons of other forms and dimensions is obtained from the purchaser, it is recommended that the tension test specimens and coupons for tension tests be prepared in accordance with the Tentative Recommended Practice for Preparing Tension Test Specimens for Copper-Base Alloy Castings (A.S.T.M. Designation: B 208),[1] except that Figs. 1, 3 and 4 of B 208 are not applicable to these alloys."

Figures 1 and 2.—Delete the present Figs. 1 and 2.

Tentative Specifications for Aluminum-Bronze Sand Castings (B 148 – 45):

Section 9.—Revise to read as follows:

9. (*a*) Unless special authority to use coupons of other forms and dimensions is obtained from the purchaser, it is recommended that the tension test specimens and coupons for tension tests be prepared in accordance with the Tentative Recommended Practice for Preparing Tension Test Specimens for Copper-Base Alloy Castings (A.S.T.M. Designation: B 208),[1] except that Figs. 1, 3, and 4 are not applicable to these alloys.

(*b*) In the case of coupons cast separately, they shall be molded in a manner similar to the castings they represent. If the castings are heat-treated, the coupons representing such castings shall be similarly heat-treated. The coupon representing alloys 9 B – HT, 9 C – HT, and 9 D – HT shall be heat-treated with the casting.

Figures 1 and 2.—Delete the present Figs. 1 and 2.

Tentative Specifications for Leaded Nickel-Brass (Leaded Nickel-Silver and Leaded Nickel-Bronze (Leaded

Nickel-Silver) Sand Castings (B 149–44 T):

Section 9 and Figs. 1 and 2.—Make the same changes as recommended above for Specifications B 143.

Tentative Specifications for Aluminum Bronze Rods, Bars, and Shapes (B 150 - 46 T):

Section 4.—Change the requirements for chemical composition to read as follows:

	Type I	Type II
Copper, per cent	80.00 to 93.00	78.00 to 85.00
Aluminum, per cent	6.50 to 11.00	9.00 to 11.00
Iron, per cent	4.00 max.	2.00 to 4.00
Nickel, per cent	1.00 max.[a]	4.00 to 5.50
Silicon, max., per cent	2.25[a]	0.25
Manganese, max., per cent	1.50	1.50
Tin, max., per cent	0.60	0.20
Zinc, max., per cent	1.00	....
Tellurium, max., per cent	0.65	....
Total of elements other than those named, max., per cent	0.50	0.50

[a] When both silicon and nickel are present in the type I alloy, only one shall be in excess of 0.25 per cent.

NOTE.—Analysis shall regularly be made only for the elements specifically mentioned in the above table. If the presence of other elements is suspected, or indicated in the course of routine analysis, further analysis shall be made either chemically or spectrographically to determine that the total of these other elements is not in excess of 0.50 per cent.

Table I.—In Table I, change the tensile strength requirement for shapes from "75,000" to "70,000."

Sections 12, 13, 14, and 16.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Table VIII.—Change the title of Table VIII to read as follows: "Straightness Tolerances. Applicable to Any Longitudinal Surface or Edge."

Section 17.—Revise to read as follows: "The material shall be free from injurious defects. Cold-drawn material shall have a bright, smooth surface."

Tentative Specifications for Copper-Nickel-Zinc Alloy Rod, Bar, and Wire (B 151 - 46 T):

Sections 13, 14, 15, and 16.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Table IX.—Change the title of Table IX to read as follows: "Straightness Tolerances. Applicable to Any Longitudinal Surface or Edge."

Tentative Specifications for Copper Sheet, Strip, and Plate (B 152 - 45 T):

Table V.—In the third column of the table for slit metal change "0.182" to "0.187."

Sections 17, 19, 20, and 21.—Add a note to read as indicated above for Sections 14 to 18 of Specifications B 19.

Section 24.—Add the words "within 30 days after receipt of the material."

New Appendix.—Add an appendix to read as indicated above for Specifications B 36.

Tentative Specifications for Phosphor Bronze Wire (B 159 - 46 T):

Section 3.—Revise to read as follows:

3. The wire shall be made from materials of such purity (Note) that it will conform to the requirements of these specifications.

NOTE.—Materials conforming to the following specifications are satisfactory for use in the manufacture of the phosphor bronze wire described in these specifications:

Material	A.S.T.M. Designation[a]
Copper	B 4, B 5, B 115, or B 216
Tin	equal to "Banca" or "Straits"

Clean, new scrap resulting from manufacturing operations upon material conforming to these specifications or clean scrap composed of material of equal purity may be used.

[a] These designations refer to the following specifications:

Standard Specifications for Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (A.S.T.M. Designation: B 4),[4]

Standard Specifications for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (A.S.T.M. Designation: B 5),[4]

Standard Specifications for Electrolytic Cathode Copper (A.S.T.M. Designation: B 115),[4] and

Tentative Specifications for Fire-Refined Copper for Wrought Alloys (A.S.T.M. Designation: B 216).[4]

Table I.—In Table I add a footnote to read as follows: "For purposes of acceptance or rejection, an observed value or calculated value obtained from

analysis should be rounded off to the nearest unit in the last right-hand place of figures, used in expressing the specified limit in accordance with the rounding-off procedure prescribed in Section 4 of the Tentative Recommended Practices for Designation of Numerical Requirements in Standards (A.S.T.M. Designation: E 29).[4]"

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[4] 1946 Book of A.S.T.M. Standards, Part I-B.

Tables II and III.—In Table II and III, add a footnote to read as follows: "For purposes of acceptance or rejection, an observed value or calculated value obtained from the tension test should be rounded off to the nearest 1000 psi. in accordance with the rounding-off procedure prescribed in Section 4 of the Tentative Recommended Practices for Designation of Numerical Requirements in Standards (A.S.T.M. Designation: E 29).[4]"

Section 10.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Tentative Specifications for Aluminum Bronze Sheet and Strip (B 169 – 44 T):

Table IV.—In the third column of the table for slit metal change "0.182" to "0.187."

Sections 10, 11, 12, and 13.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Section 16.—Add the words "within 30 days after receipt of the material."

New Appendix.—Add an appendix as indicated above for Specifications B 36.

Tentative Specifications for Copper Bus Bars, Rods, and Shapes (B 187 – 45 T):

Sections 11, 12, 13, and 14.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Section 13.—Revise to read as follows: "Unless otherwise specified, the material shall be furnished in straight lengths. The deviation from absolute straightness of any longitudinal surface or edge shall not exceed the limitations prescribed in Table VII."

Table VII.—Revise Table VII to read as shown in the accompanying Table I:

TABLE I. STRAIGHTNESS TOLERANCES. Applicable To Any Longitudinal Surface Or Edge.

	Maximum Curvature (depth of arc), in.	Portion of Total Length in Which Depth of Arc is Measured, in.
Drawn rods	$\frac{1}{2}$	120
Drawn shapes	$\frac{1}{2}$	72
Drawn bars	$\frac{1}{4}$	60

Section 18.—Revise to read as follows: "Material that fails to conform to these specifications will be rejected and the manufacturer shall be notified within 30 days after receipt of the material."

Tentative Specifications for Copper Bus Pipes and Tubes (B 188 – 45 T):

Sections 11 and 12.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Table III.—In Table III, delete the word "Standard" from the title.

Section 16.—Revise to read as follows: "Material that fails to conform to these specifications will be rejected, and the manufacturer shall be notified within 30 days after receipt of the material."

Tentative Specifications for Beryllium-Copper Alloy Strip (B 194 – 46 T):

Table V.—In the second column of Table V change "0.182" to "0.187."

Sections 12, 13, 14, and 15.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

New Appendix.—Add an appendix as indicated above for Specifications B 36.

Tentative Specifications for Beryllium-Copper Alloy Strip, Special Grade (B 195 – 45 T):

Sections 12, 13, 14, and 15.—Add a note to read as indicated above for Sections 14 to 18 of Specifications B 19.

Section 18.—Add the words "within 30 days after receipt of the material."

Tentative Specifications for Beryllium-Copper Alloy Rod and Bar (B 196 - 46 T):

Sections 12, 13 and 14.—Add a note to read as indicated above for Sections 14 to 18 of Specifications B 19.

Table XII.—Change the title for Table XII to read as follows: "Straightness Tolerances. Applicable to Any Longitudinal Surface or Edge."

Tentative Specifications for Beryllium-Copper Alloy Wire (B 197 - 46 T):

Section 4.—Add a footnote to the table in this section to read as indicated above for Table I of Specifications B 159.

Tables II and III.—Add a footnote to Tables II and III to read as indicated above for Tables II and III of Specifications B 159.

Section 12.—Change the title to "Dimensional Tolerances" and add a note as indicated above for Sections 14 to 18 in Specifications B 19.

Tentative Specifications for Silicon-Bronze and Silicon-Brass Sand Castings (B 198 - 45 T):

Section 6.—Revise Paragraph (*a*) to read as follows:

> 6. (*a*) It is recommended that the tension test specimens and coupons for tension tests be prepared in accordance with the Tentative Recommended Practice for Preparing Tension Test Specimens for Copper-Base Alloy Castings (A.S.T.M. Designation: B 208),[1] except that Fig. 2 of B 208 is not applicable to these alloys.
>
> (*b*) Test specimens may be chilled provided the castings are also chilled in the same manner.

Reletter Paragraph (*b*) as (*c*), omit present Paragraph (*c*), and retain Paragraph (*d*).

Figures 1 and 2.—Delete the present Figs. 1 and 2.

Tentative Specifications for Copper-Nickel-Zinc Alloy Wire (B 206 - 46 T):

Section 3.—In line 2 of Paragraph (*a*) delete the word "raw." Insert the present Paragraph (*b*) as a part of the note under Paragraph (*a*) and reletter the present Paragraph (*c*) as (*b*).

Sections 12 and 14.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends, for immediate adoption, revisions in 25 specifications as indicated below and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these modifications may be referred to letter ballot of the Society.

Standard Specifications for Copper Plates for Locomotive Fireboxes (B 11 - 41):

Section 14.—Revise to read as follows: "Material that fails to conform to these specifications will be rejected, and the manufacturer shall be notified within 30 days after receipt of the material."

Standard Specifications for Copper Rods for Locomotive Staybolts (B 12 - 45):

Section 5.—Revise to read as follows: "An analysis may be made by the purchaser from samples selected according to Section 10. The chemical composition thus determined shall conform to the requirements specified in Section 4."

Section 9 (*a*).—Revise to read as follows: "Two tension and two bend tests shall be made on each lot of 10,000 lb. or fraction thereof on samples selected according to Section 10."

New Section.—Add a new Section 10 to read as follows, renumbering the subsequent sections accordingly:

> 10. *Sampling.*—Samples from separate pieces shall be taken from 0.5 per cent of the number of pieces in each lot of 10,000 lb. or fraction thereof, but in no case shall less than 4 nor more than 20 samples be taken.

Section 11.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Standard Specifications for Free-Cutting Brass Rod and Bar For Use in Screw Machines (B 16 – 45):

Section 9.—Reletter as Paragraph (*a*) and add the following as Paragraph (*b*): "(*b*) Bend test specimens may be a piece of the full-size rod or a piece 1 in. in width by ½ in. in thickness. In the case of bend test specimens of rectangular section, the edges may be rounded off to a radius equal to one-fourth of the thickness."

Sections 12, 13, 14, and 15.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Table VI.—Change the title for Table VI to read as follows: "Straightness Tolerances. Applicable to Any Longitudinal Surface or Edge."

Section 16.—Revise to read as follows: "16. The material shall be free from injurious defects and shall have a bright, smooth surface."

Standard Specifications for Copper Pipe, Standard Sizes (B 42 – 45):

Section 7.—Reletter as Paragraph (*a*) and add the following as Paragraph (*b*): "(*b*) As an alternate to the expansion test for pipe over 4 in. in diameter in the annealed condition, a section 4 in. in length shall be cut from the end of one of the lengths for a flattening test. This 4-in. specimen shall be flattened so that a gage set at three times the wall thickness will pass over the pipe freely throughout the flattened part. The pipe so tested shall develop no cracks or flaws visible to the unaided eye as a result of this test. In making the flattening test the elements shall be slowly flattened by one stroke of the press."

Section 8.—Revise last sentence to read as follows: "This test shall apply only to sizes 2 in. and under in outside diameter."

Sections 13 and 14.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Section 14 (a).—Revise to read as follows: "14. (*a*) The recommended standard length of round seamless pipe shall be 12 ft. The length shall conform to the tolerances prescribed in Table III."

Section 17.—Change the title "Rejection" to "Retests."

New Section.—Add a new Section 18 to read as follows: "18. *Rejection.*—Material that fails to conform to these specifications will be rejected, and the manufacturer shall be notified within 30 days after receipt of the material."

Standard Specifications for Brass Pipe, Standard Sizes (B 43 – 45):

Title.—Change the title to read "Standard Specifications for Red Brass Pipe, Standard Sizes."

Section 1.—Revise to read as follows: "These specifications cover seamless red brass pipe in all standard pipe sizes. . . ."

Section 2.—Delete item (*1*) and renumber the remaining items accordingly.

Section 4.—In the third sentence delete the words "in the case of red brass pipe only."

Section 5.—Delete columns headed "Muntz Metal," "High Brass," and "Admiralty Metal" from the table of chemical requirements.

Section 7.—Add a new Paragraph (*b*) to read as indicated above for Section 7 (*b*) of Standard Specifications B 42.

Section 9.—Delete the last sentence and replace by the following: "This test shall apply only to sizes 2 in. and under in outside diameter."

Table I.—In Table I delete the column headed "Yellow Brass" and revise the table title accordingly.

Sections 14 and 15.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Section 15 (a).—Revise to read as follows: "15. (*a*) The recommended standard length of round seamless pipe shall be 12 ft. The lengths shall conform to the tolerances prescribed in Table III."

Section 19.—Change the title "Rejection" to "Retests."

New Section.—Add a new Section 20 to read as follows: "20. *Rejection.*—Material that fails to conform to these specifications will be rejected, and the manufacturer shall be notified within 30 days after receipt of the material."

Standard Specifications for Phosphor Copper (B 52 – 43):

Section 3.—Revise by the addition of the following words in parentheses after the word "pounds": "(or in the form of shot)."

Section 5.—Revise to read as follows:

5. (*a*) *Ingots or Slabs.*—For ingots or slabs, approximately 1 lb. of material for each 100 lb. of the lot shall be selected as the sample. The sample shall be examined for extraneous material. Depending on the grade, the sampling shall then be carried out as follows:

(*1*) *Grade A.*—The sample shall be crushed and quartered. From the quarter selected for analysis sufficient material shall be reduced to suitable size particles by means of a bucking board (hardened steel plate with a curved-face mallet). A magnet shall be passed through the sample to insure the elimination of free iron.

(*2*) *Grade B.*—The entire sample, or a sample taken by quartering if the shipment is a large one, shall be melted under a layer of charcoal. The melted sample shall be cast into a small ingot or button. The ingot or button shall be drilled or sawed completely through, or one-half through from the top and bottom, surface material being rejected.

(*b*) *Shot.*—For material in shot form a representative 1-lb. sample shall be selected. Care shall be exercised to have the coarse material and fine material in the sample in approximately the same proportions in which they exist in the lot. The sample shall be examined for extraneous material, which shall be removed. The selected sample shall be quartered and a magnet passed through the selected quarter to insure the elimination of free iron.

Standard Specifications for Silicon Copper (B 53 – 43):

Section 3.—Add the word "shot" before the word "lumps."

Section 4.—Revise the chemical requirements for silicon and iron to read as follows:

	Grade A	Grade B	Grade C
Silicon, per cent	10.0 to 12.0	18.5 to 21.5	28.5 to 31.5
Iron, max., per cent	0.50	0.50	0.50

Add limits for calcium, as follows:

Calcium, max, per cent	0.15	0.15	0.15

Section 5.—Revise to read as follows:

5. (*a*) *Ingots or Slabs.*—For ingots or slabs, approximately 1 lb. of material for each 100 lb. of the lot shall be selected as the sample. The sample shall be examined for extraneous material. The sample shall be crushed and quartered. From the quarter selected for analysis sufficient material shall be reduced to suitable size particles by means of a bucking board (hardened steel plate with a curved-face mallet). A magnet shall be passed through the sample to insure the elimination of free iron.

(*b*) *Shot or Lumps.*—For material in shot or lump form a representative 1-lb. sample shall be selected. Care shall be exercised to have the coarse material and fine material in the sample in approximately the same proportions in which they exist in the lot. The sample shall be examined for extraneous material, which shall be removed. The selected sample shall be quartered and a magnet passed through the selected quarter to insure the elimination of free iron.

Standard Specifications for Steam or Valve Bronze Castings (B 61 – 44):

Section 8.—Revise to read as follows: "8. It is recommended that the tension test specimens and coupons for tension tests be prepared in accordance with the Tentative Recommended Practice for Preparing Tension Test Specimens for Copper-Base Alloy Castings (A.S.T.M. Designation: B 208),[1] except that Fig. 2 of B 208 is not applicable to this alloy."

Figures 1 and 2.—Delete the present Figs. 1 and 2.

Standard Specifications for Composition Brass or Ounce Metal Castings (B 62 – 44):

Section 8.—Revise to read as indicated for Section 8 of Specifications B 61.

Figures 1 and 2.—Delete the present Figs. 1 and 2.

Standard Specifications for Bronze Castings in the Rough for Locomotive Wearing Parts (B 66 – 44):

Table I.—In Table I add a decimal point followed by a zero for all values of tin and lead.

Standard Specifications for Car and Tender Journal Bearings, Lined (B 67 – 44):

Section 3.—Add a decimal point followed by a zero for all values in chemical composition where no decimals are now shown.

Standard Specifications for Seamless Copper Tubing, Bright Annealed (B 68 – 45):

Section 8.—Reletter as Paragraph (*a*) and add a new Paragraph (*b*) as indicated above for Tentative Specifications B 75.

Sections 13 and 14.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Table I.—Delete the column headed "Stubs' Gage."

Section 18.—Revise to read as follows: "18. Material that fails to conform to these specifications will be rejected, and the manufacturer shall be notified within 30 days after receipt of the material."

Standard Specifications for Copper Water Tube (B 88 – 45):

Section 9.—Reletter as Paragraph (*a*) and add a new Paragraph (*b*) as indicated above for Section 8 (*b*) of Tentative Specifications B 75.

Section 11.—Change the last line of Paragraph (*a*) to read "specified in Sections 8 to 10, inclusive and Section 15 (*b*)." In the second line of Paragraph (*b*) delete "30 to 100-ft."

Table II.—Delete the $\frac{1}{8}$-in. and $\frac{1}{4}$-in. sizes, and for type M tubes delete all values for sizes 2 in. and under in the columns headed "wall thickness" and "theoretical weight."

Section 14 (b).—Revise to read as follows: "(*b*) The nominal length for tubes furnished in coils shall be 60 ft."

Sections 15 and 16.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Section 15 (b).—Change to read: "(*b*) No single tube as selected in Section 11 shall vary . . . ."

Standard Specifications for Copper-Silicon Alloy Plate and Sheet for Pressure Vessels (B 96 – 42):

Section 9.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Section 14.—Add the words "within 30 days after receipt of the material."

Standard Specifications for Copper-Silicon Alloy Sheet and Strip for General Purposes (B 97 – 44):

Table V.—In the third column of the table for slit metal change "0.182" to "0.187."

Sections 10, 11, 12, and 13.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Section 16.—Add the words "within 30 days after receipt of the material."

New Appendix.—Add a new Appendix II to read as indicated above for Specifications B 36.

Standard Specifications for Copper-Silicon Alloy Rods, Bars, and Shapes (B 98 – 45):

Table II.—In Table II revise the heading "Types A, C, and D alloy rods and shapes" to read "Types A, C, and D alloy rods, square bars and shapes"; also

in the first part of the table, for half-hard and hard tempers, revise "rods" to read "rods and square bars."

Sections 11, 12, 13, and 14.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Table VIII.—Change the title for Table VIII to read as follows: "Straightness Tolerances. Applicable to Any Longitudinal Surface or Edge."

Section 15.—Revise to read as follows: "15. The material shall be free from injurious defects, and, unless otherwise specified, shall have a bright, smooth surface."

Standard Specifications for Copper-Silicon Alloy Wire for General Purposes (B 99 – 45):

Table I.—Add a footnote to read as indicated above for Table I of Tentative Specifications B 159.

Table II.—Add a footnote to read as indicated above for Tables II and III of Specifications B 159.

Section 6.—Delete Paragraph (*b*).

Section 7.—Revise the first two sentences to read as follows: "Wire of types A, B, and C alloys shall stand wrapping one full turn (360 deg.) around its own diameter without fracture. This test shall not apply to wire over ½ in. in diameter nor to material of type D."

Section 11.—Delete Paragraphs (*a*) and (*c*) and add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Section 12 (b).—Revise to read as follows: "(*b*) Necessary brazes in the wire shall be made prior to final drawing and in accordance with best commercial practice."

Standard Specifications for Rolled Copper-Alloy Bearing and Expansion Plates for Bridge and Other Structural Uses (B 100 – 44):

Section 8.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Section 11.—Revise to read as follows: "11. Material that fails to conform to these specifications will be rejected, and the manufacturer shall be notified within 30 days after receipt of the material."

Standard Specifications for Phosphor Bronze Sheet and Strip (B 103 – 44):

Table I.—Add "grade B 1" with composition as for grade B 1 in Tentative Specifications B 139.

Table II.—Change the title to include grade B 1 with the same properties as for grade B.

Table IV.—In the third column of the table for slit metal change "0.182" to "0.187."

Sections 13, 14, 15, and 16.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Section 19.—Add the words "within 30 days after receipt of the material."

New Appendix.—Add an appendix to read as indicated above for Specifications B 36.

Standard Specifications for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 45):

Table I.—Delete "aluminum brass, type A" and add a footnote to read "type A aluminum brass has been discontinued."

Section 9.—Add a sentence to read as follows: "The flattened elements shall not show cracking or rupture clearly visible to the unaided eye."

Section 14.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Standard Specifications for Copper-Base Alloy Forging Rods, Bars, and Shapes (B 124 – 45):

Title.—Revise to read "Copper and Copper-Base Alloy Forging Rods, Bars, and Shapes."

Sections 6 and 7.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Section 10—Add the words "within 30 days after receipt of the material."

Standard Specifications for Brass Wire (B 134 – 45):

Section 1.—Delete alloy No. 5 and add a footnote to read, "The former alloy No. 5 has been discontinued."

Section 3.—Revise and combine Paragraphs (*a*) and (*b*) as a new Paragraph (*a*) to read as follows:

> 3. (*a*) The wire shall be made from wire of such purity (Note) that it will conform to the requirements of these specifications.
>
> NOTE.—Materials conforming to the following specifications are satisfactory for use in the manufacture of the brass wire described in these specifications:
>
> | Material | A.S.T.M. Designation[a] |
> |---|---|
> | Copper | B 4, B 5, B 115, or B 216 |
> | Zinc | B 6 |
>
> Clean new scrap resulting from manufacturing operations upon material complying with these specifications or clean scrap composed of material of equal purity may be used.

Table I.—Revise the copper limits for alloy No. 2 to read "89.0 to 91.0" and add decimal points followed by a zero for all values in chemical composition where no decimals are now shown. Delete alloy No. 5 and add a footnote to read "The former alloy No. 5 has been discontinued." Add a footnote to read as indicated above for Table I of Specifications B 159.

Table II.—In Table II add a footnote to read as indicated above for Tables II and III of Specifications B 159.

Sections 12 and 14.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Standard Specifications for Manganese Bronze Rods, Bars, and Shapes (B 138 – 45):

Sections 12, 13, 14, and 16.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Table IX.—Change the title of Table IX to read as follows: "Straightness Tolerances. Applicable to Any Longitudinal Surface or Edge."

Section 15.—Revise to read as follows: "15. The material shall be free from injurious defects. Cold-drawn material shall have a bright, smooth surface."

Standard Specifications for Leaded Red Brass (Hardware Bronze) Rods, Bars, and Shapes (B 140 – 45):

Table I.—Revise Table I to read as shown in the accompanying Table II.

Sections 9, 10, 11, and 12.—Add a

TABLE II.—TENSILE REQUIREMENTS.

Temper	Forms	Diameter or Thickness, in.	Width, in.	Tensile Strength, min., psi.	Yield Strength, min., psi.	Elongation in 2 in., min., per cent
Soft	All forms	All sizes	....	35 000	10 000	25.0
Half hard	Rounds, hexagons, octagons	0.5 and under		50 000	30 000	8.0
		Over 0.5 to 1.0		45 000	27 000	10.0
		Over 1.0		40 000	25 000	15.0
	Rectangles and squares	1.0 and under	2.0 and under	45 000	25 000	10.0
		Over 1.0	Over 2.0	40 000[a]	15 000[a]	20.0[a]

[a] In the case of bars either over 1 in. in thickness or over 2 in. in width, or both, these values shall apply.

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[a] These designations refer to the following specifications:
Standard Specifications for Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (A.S.T.M. Designation: B 4),[4]
Standard Specifications for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (A.S.T.M. Designation: B 5),[4]
Standard Specifications for Electrolytic Cathode Copper (A.S.T.M. Designation: B 115),[4]
Tentative Specifications for Fire-Refined Copper for Wrought Alloys (A.S.T.M. Designation: B 216),[4] and
Standard Specifications for Slab Zinc (Spelter) A.S.T.M. Designation: B 6).[4]

note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Table VI.—Change the title of Table VI to read as follows: "Straightness Tolerances. Applicable to Any Longitudinal Surface or Edge."

Section 12.—Revise to read as follows: "The material shall be free from injurious defects, and shall have a clean, smooth surface."

Standard Specifications for Copper-Alloy Condenser Tube Plates (B 171–45):

Sections 9, 10 and 11.—Add a note to read as indicated above for Sections 14 to 18 in Specifications B 19.

Section 15.—Add the words "within 30 days after receipt of the material."

ADOPTION OF TENTATIVE REVISIONS OF STANDARDS AS STANDARD

The committee recommends the adoption as standard of the tentative revisions issued in 1946 in the following nine standard specifications:

Standard Specifications for Copper Rods for Locomotive Staybolts (B 12 – 45),

Standard Specifications for Free-Cutting Brass Rod and Bar for Use in Screw Machines (B 16 – 45),

Standard Specifications for Copper Water Tube (B 88 – 45),

Standard Specifications for Copper-Silicon Alloy Rods, Bars, and Shapes (B 98 – 45),

Standard Specifications for Copper-Silicon Alloy Wire for General Purposes (B 99 – 45),

Standard Specifications for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 45),

Standard Specifications for Brass Wire (B 134 – 45),

Standard Specifications for Manganese Bronze Rods, Bars, and Shapes (B 138 – 45), and

Standard Specifications for Leaded Red Brass (Hardware Bronze) Rods, Bars, and Shapes (B 140 – 45).

REPORT OF COMMITTEE B-6*

ON

DIE-CAST METALS AND ALLOYS

Committee B-6 on Die-Cast Metals and Alloys did not hold any meetings during the year because of traveling restrictions and inadequate hotel accommodations.

A balance of $2,456.68 was reported by Society Headquarters in the funds of Committee B-6 as of December 31, 1945.

At the present time, the committee consists of 83 members, of whom 38 are classified as producers, 23 as consumers, and 22 as general interest members.

I. Revision of Tentative

The committee recommends the following revision in the Tentative Specifications for Aluminum-Base Alloy Die Castings (B 85 – 44 T):[1]

Section 1.—Change the second sentence to refer to nine alloys and add references to alloys SG2 and SG3.

Table I.—Add the following requirements on chemical composition for the two new alloys:

	Alloy SG2	Alloy SG3
Copper, max., per cent	0.6	0.6
Iron, max., per cent	1.3	2.0
Silicon, per cent	9.0 to 10.0	9.0 to 10.0
Manganese, max., per cent	0.3	0.3
Magnesium, per cent	0.4 to 0.6	0.4 to 0.6
Zinc, max., per cent	0.5	0.5
Nickel, max., per cent	0.5	0.5
Tin, max., per cent	0.1	0.1
Other constituents, except aluminum, max., (total) per cent	0.2	0.2
Aluminum, per cent	remainder	remainder

Also change footnote *b* under Table I to read as follows: "Alloys S4, S5, SC2, SC7 and SG3 are suitable for the production of castings by either the gooseneck or the cold-chamber process. Castings of alloys S9, SC5, SC6 and SG2 may be made only in cold-chamber machines."

Appendix.—Add the following data on mechanical properties of the two new alloys:

	Alloy SG2	Alloy SG3
Tensile strength (round specimens), psi	43 000	43 000
Elongation in 2 in. (round specimens), per cent	5	3
Charpy Impact (square specimens), ft-lb	4.2	2.7

II. Revision of Standard, Immediate Adoption

The committee recommends for immediate adoption the following revision in the Standard Specifications for Zinc-Base Alloy Die Castings (B 86 – 43)[1] and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that this recommendation may be referred to letter ballot of the Society:

New Section.—Add the following new Section 6, renumbering the remaining sections accordingly:

6. *Chemical Analysis.*—The chemical analyses of the die castings shall be made in accordance with the Tentative Methods of Spectrochemical Analysis of Zinc-Alloy Die Castings for Minor Constituents and Impurities (A.S.T.M. Designation: E 27) and with the Tentative Methods of Chemical Analysis of Zinc-Base Die-Casting Alloys (A.S.T.M. Designation: E 47) for the major constituents.

Sections 7 and 8.—Omit these sections

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] 1944 Book of A.S.T.M. Standards, Part I.

on stability and expansion tests, respectively.

III. WITHDRAWAL OF TENTATIVE SPECIFICATIONS

The committee recommends the withdrawal of the Tentative Specifications for Special Quality Zinc-Base Alloy Die Castings (B 186 – 42 T).[1]

IV. TENTATIVE SPECIFICATIONS CONTINUED AS TENTATIVE

The committee recommends that the Tentative Specifications for Magnesium-Base Alloy Die Castings (B 94 – 44 T),[1] and the Tentative Specifications for Copper-Base (Brass) Alloy Die Castings (B 176 – 42 T)[1] be continued as tentative without revision.

V. EMERGENCY SPECIFICATIONS DISCONTINUED

The committee recommends that the Emergency Specifications for Special Quality, Aluminum-Base Alloy Die Castings (ES – 29) and the Emergency Specifications for Special Quality, Magnesium-Base Alloy Die Castings (ES – 40) be discontinued.[2]

The recommendations appearing in this report have been submitted to letter ballot of the committee, which consists of 83 members; 75 members returned their ballots with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum-Base Die-Casting Alloys (J. J. Bowman, chairman) started atmospheric exposure tests on test bars of alloys G2 (8 per cent magnesium, remainder aluminum) and SG3 (9.5 per cent silicon, 0.5 per cent magnesium, remainder aluminum) in November, 1944, at Sandy Hook, N. J., and New York, N. Y.

Unfortunately at the end of the 1-yr. exposure period, it was discovered that most of the G2 specimens at Sandy Hook were missing. However, the sea coast exposure test of this alloy was salvaged by means of some extra specimens which were being held by the Bell Telephone Laboratories for emergency purposes. On March 6, 1946, a new set of bars from the original lot was placed in the Sandy Hook rack. The few remaining test specimens from the originally exposed set were removed from the rack to avoid future confusion.

The testing results on the two alloys from the New York exposure and on the one alloy from Sandy Hook, after 1-yr. of atmospheric exposure, are appended hereto.

Subcommittee II on Zinc-Base Die-Casting Alloys (A. E. Weiss, chairman) recommended the discontinuance of Tentative Specifications for Special Quality Zinc-Base Alloy Die Castings (B 186 – 42 T).

Subcommittee III on Tin and Lead-Base Die Casting Alloys (G. O. Hires, chairman) submitted data on creep tests on tin and lead alloys. These results are appended hereto.

Subcommittee V on Exposure and Corrosion Tests (W. M. Peirce, chairman) completed the testing of the aluminum-base and zinc-base alloys which were exposed for 15 yr. at five outdoor and four indoor sites. The results of inspection and tests of these alloys after 1, 5, and 10-yr. exposures were included in the 1932, 1935, and 1940 reports,[3] respectively.

The committee also tested the magnesium-base and zinc-base alloys from five outdoor and four indoor sites after 5 yr. of exposure. The record of the original properties of these alloys was included in the 1939 report.[4]

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[1] See Editorial Note, p. 224.

[3] Report of Subcommittee V on Exposure and Corrosion Test of Committee B-6 on Die-Cast Metals and Alloys, *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part I, pp. 265, 271 (1932), Vol. 35, Part I, p. 190 (1935), and Vol. 40, p. 195 (1940).

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 280 (1939).

All of the above data appear in the report of this subcommittee appended hereto.

A nominating committee consisting of G. O. Hiers, R. L. Templin, and A. E. Martin was appointed to present nominees for chairman, vice-chairman, and secretary at the June meeting. The report of the nominating committee was acted upon by the committee and resulted in the re-election of the present officers for the ensuing term of two years.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. REVISION OF TENTATIVE			
Spec. for Aluminum-Base Alloy Die Castings (B 85 – 44 T)......	47	0	18
II. REVISION OF STANDARD, IMMEDIATE ADOPTION			
Spec. for Zinc-Base Alloy Die Castings (B 86 – 43)...........	47	1	17
III. WITHDRAWAL OF TENTATIVE SPECIFICATIONS			
Spec. for Special Quality Zinc-Base Alloy Die Castings (B 186 – 42 T)................	53	0	22
IV. TENTATIVE SPECIFICATIONS CONTINUED AS TENTATIVE			
Spec. for Magnesium-Base Alloy Die Castings (B 94 – 44 T).....	59	0	16
Spec. for Copper-Base (Brass) Alloy Die Castings (B 176 – 42 T)........................	50	0	25

This report has been submitted to letter ballot of the committee, which consists of 83 members; 75 members returned their ballots, of whom 71 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. R. TOWNSEND,
Chairman.

G. L. WERLEY,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-6 presented to the Society through the Administrative Committee on Standards the recommendation that the Emergency Specifications for Special Quality, Magnesium-Base Alloy Die Castings (ES – 40), and the Emergency Specifications for Special Quality Aluminum-Base Alloy Die Castings (ES – 29) be withdrawn. This recommendation was accepted by the Standards Committee on September 9, 1946.

REPORT OF SUBCOMMITTEE I ON ALUMINUM-BASE DIE-CASTING ALLOYS

AN INVESTIGATION OF ALUMINUM DIE-CASTING ALLOYS NOS. IVa, Va, AND V

The 1935 report[1] of Subcommittee I described the preparation of the specimens for this investigation and presented the results of chemical analyses and "original" mechanical properties tests. The following year the results of the accelerated corrosion tests were published with the general conclusion that, although the differences were not large, the resistance of alloy No. Va to severely corrosive conditions was superior to that of alloys Nos. IVa and V. Visual and microscopic examinations of flat specimens were made for the first time in 1939 after 4-yr. exposure to the atmosphere.[2] The first set of round specimens and the second set of flat specimens were removed from the atmospheric exposure racks after a period of 5 yr. The results[3] disclosed small differences among the several materials.

The present report describes the examination and tests of specimens removed from the atmospheric racks after an exposure of 10 yr. The results show that, on the average, the losses in tensile strengths were small, but significant, and were only slightly greater than those obtained after 5-yr. exposure. On the average, no really significant differences existed among the three alloys tested or between the same alloy cast by two different suppliers.

Description:

Since Table I of the 1935 report details the distribution of specimens in the tests, it will suffice here merely to say that three racks, each containing 180 round tension specimens and 60 flat tension specimens were exposed at the Sandy Hook, N. J., New York, N. Y, and Altoona, Pa. test sites. In accordance with the practice established by Subcommittee V, the round specimens were exposed with the axis in a horizontal plane. The flat specimens, since they were to be used for studying the rate of surface attack, were exposed so that one flat surface was horizontal.

At the end of 10-yr. exposure, as in the case of the 5-yr. period, 60 round tension specimens and 12 of the flat specimens were removed from each rack for test and examination. Missing from the test racks at this period were 4 round specimens and 9 flat specimens at Altoona and 3 flat specimens at New York. Since there are two lots of three alloys in the test, this generally provided 10 round and 2 flat specimens of each material. Five of the round specimens of each material were tested by the Hawthorne Plant of the Western Electric Co. and the other five were tested by the Aluminum Research Laboratories. Each laboratory tested five blanks of each material taken from a group that had been stored indoors for 10 yr. Consistent with previous methods of testing,

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[1] "An Investigation of Aluminum Die-Casting Alloys Nos. IVa and Va," Appendix I, Report of Committee B-6 on Die-Cast Metals and Alloys, *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part I, p. 184 (1935); Vol. 36, Part I, p. 182 (1936).

[2] Report of Committee B-6 on Die-Cast Metals and Alloys, *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 278 (1939).

[3] Report of Committee B-6 on Die-Cast Metals and Alloys, *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 191 (1940).

The accompanying Table A showing the chemical analyses of the three alloys is reproduced from the 1935 Report.[1] The results summarized in Table A are believed to represent the typical compositions of the materials in the test.

TABLE A.—CHEMICAL ANALYSES OF MATERIALS.

Identification Number	Alloy	Silicon, per cent	Iron, per cent	Copper, per cent	Manganese, per cent	Magnesium, per cent	Zinc, per cent	Nickel, per cent	Tin, per cent	Lead, per cent	Aluminum, per cent
ROUND SPECIMENS[a]											
14865	No. IVa	4.72	1.06	0.10	0.01	0.01	0.00	0.01	0.00	0.00	balance
15431	No. IVa	4.78	1.11	0.10	0.02	0.01	0.00	0.01	0.00	0.00	balance
14866	No. Va	12.02	1.06	0.09	0.01	0.01	0.00	0.01	0.00	0.00	balance
15432	No. Va	11.87	1.34	0.09	0.01	0.00	0.00	0.01	0.00	0.00	balance
14867	No. V	12.18	1.37	0.42	0.25	0.05	0.51	0.31	0.05	0.02	balance
15433	No. V	12.09	1.51	0.42	0.26	0.06	0.52	0.32	0.05	0.03	balance
FLAT SPECIMENS[b]											
14865	No. IVa	4.75	1.07	0.09	0.01	0.01	0.00	0.01	0.00	0.00	balance
15431	No. IVa	4.67	1.09	0.10	0.02	0.00	0.00	0.01	0.00	0.00	balance
14866	No. Va	12.13	1.11	0.09	0.01	0.01	0.00	0.02	0.00	0.00	balance
15432	No. Va	11.91	1.22	0.09	0.01	0.00	0.00	0.01	0.00	0.00	balance
14867	No. V	12.16	1.40	0.42	0.24	0.05	0.51	0.31	0.05	0.03	balance
15433	No. V	12.01	1.51	0.42	0.26	0.06	0.52	0.32	0.05	0.03	balance
			max.	max.	max.	max.	max.	max.	max.	max.	
Nominal	No. IVa	5.0	1.5	0.15	0.1	0.05	0.20	0.10	0.05	0.05	balance
	No. Va	12.0	1.5	0.15	0.1	0.05	0.20	0.10	0.05	0.05	balance
	No. V	12.0	2.0	0.6	0.3	0.1	0.75	0.50	0.10	....	....

[a] Composite from 5 specimens.

[b] Composite from 2 specimens.

TABLE I.—INVESTIGATION OF ALUMINUM DIE-CASTING ALLOYS IVa AND Va—RESULTS OF EXPOSURE FOR 10 YR. TO THE ATMOSPHERE.

Alloy[a]	Original Properties[b]			Sandy Hook, N. J.[c]			New York, N. Y.[c]			Altoona, Pa.[c]		
				Percentage Change			Percentage Change			Percentage Change		
	Tensile Strength	Yield Strength	Elongation	Tensile Strength	Yield Strength	Elongation	Tensile Strength	Yield Strength	Elongation	Tensile Strength	Yield Strength	Elongation
TESTED BY ALUMINUM RESEARCH LABORATORIES												
No. 14865 (IVa)	26 700	10 740	5.5	−1.3	+3.2	+1.8	−4.5	−2.0	−18.2	−5.9[e]	−0.7[e]	0.0[e]
No. 15431 (IVa)	25 900	12 880	4.8	−4.4	+0.8	−4.2	−1.9	+2.0	−20.8	−4.0[e]	−0.4[e]	0.0[e]
No. 14866 (Va)	34 260	16 960	2.7	−7.3	+1.6	−1.5	−4.7	−1.5	−14.8	−6.2	+0.7	−11.1
No. 15432 (Va)	26 000	16 680	1.5	−0.7	+3.2	+6.3	−0.9	+3.4	−6.7	−3.6	+0.6	+11.8
No. 14867 (V)	35 980	20 460	1.9	−2.7	−5.4	−5.3	−5.8	−6.0	−5.3	−6.8	−8.4	+20.8
No. 15433 (V)	30 760	20 640	1.3	−11.0	−7.4	−10.0	−6.9	−5.6	−7.1	−16.5	−9.7	0.0
TESTED BY WESTERN ELECTRIC CO.												
No. 14865 (IVa)	28 482	11 612	5.6	−3.5	+2.2	0.0	−5.8	−1.9	−8.9	−8.3	−5.1	−12.5
No. 15431 (IVa)	27 376	14 008	4.3	−2.7	−5.2	+11.6	−8.5	−7.0	−7.0	−5.6	−7.8	+2.3
No. 14866 (Va)	33 695[d]	17 628[d]	1.5[d]	+3.9	−0.7	+33.0	−6.5	−4.8	0.0	−0.2	−1.7	−13.3
No. 15432 (Va)	28 990	18 457	1.1	−10.2	−6.3	0.0	−8.2	−1.9	+18.2	−9.1	−8.7	+27.3
No. 14867 (V)	37 756	20 698	1.4	−2.9	−4.5	−7.1	−7.6	−3.5	+7.1	−4.5	−4.1	0.0
No. 15433 (V)	29 984	19 970	1.0	−5.1	−0.5	−10.0	−3.4	−0.7	−20.0	+3.5	−0.5	+20.0

[a] Alloys Nos. 14865, 14866, 14867 (Nos. IVa, Va, V, respectively) were cast by the Aluminum Company of America. Alloys Nos. 15431, 15432, 15433 (Nos. IVa, Va, V, respectively) were cast by the General Electric Co.

[b] Average of five specimens stored 10 yr. indoors.

[c] Average of five exposed specimens.

[d] Average of four specimens; one specimen with obviously low mechanical properties was omitted.

[e] Average of three specimens; two were missing at the 10-yr. period.

the mechanical properties tests were made with Amsler hydraulic testing machines using the testing procedure regularly employed for non-ferrous specimens. The yield strengths reported were determined by the aid of an automatic, autographic electrical extensometer. The visual and microscopic examinations of the flat specimens were made by the Aluminum Research Laboratories, using polished and etched transverse sections and a magnification of 100.

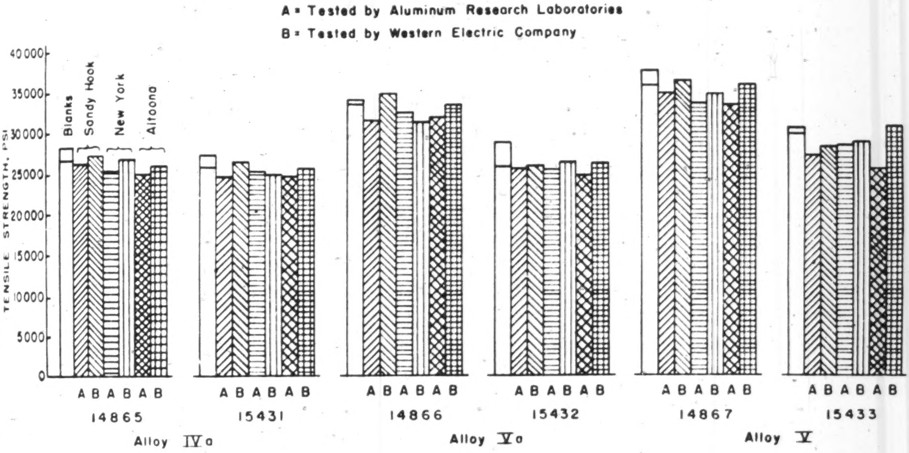

Fig. 1.—Effect of 10-yr. Exposure to the Atmosphere on the Tensile Strength of Aluminum Alloy Die Castings.

Test Results:

Table I shows the average mechanical properties of the blanks tested after 10-yr. storage indoors and the percentage changes in properties for the specimens exposed for 10 yr. at the three outdoor stations (as compared with the blanks). The tensile strengths of the specimens after 10-yr. storage (data for the two testing stations being superimposed on one another) or exposure outdoors are presented graphically in Fig. 1. Table II summarizes the changes in properties, without regard to testing station, of the specimens exposed for 10 yr. outdoors and includes for purposes of comparison similar data on the specimens exposed for 5 yr. outdoors. Two methods of calculating the changes in mechanical properties were used. The properties of the exposed specimens were compared in one case with those of the single set (10 specimens) of blanks that had been stored indoors for the corresponding period of time, and in the other case with the average of all blanks (100 specimens) tested over a 10-yr. period. The foregoing data have been further summarized in Table III, omitting changes in elongation, and including the coefficient of variation of the 10-yr. storage specimens and the 10-yr. exposure specimens. The average percentage loss in tensile strength of the sets of specimens exposed to the three atmospheric exposure stations are present in Table IV. In Table V are given the microscopic examination of the flat specimens, including the data obtained at the 5-yr. period.

The average mechanical properties of the blanks tested at various periods of time over a 10-yr. period are presented in Table VI. The dates on which the different lots of specimens had been cast, the subsequent dates on which they were tested mechanically and the approximate period of natural aging at room temperature prior to testing are indicated.

Discussion:

The data in Table I reveal that the changes in mechanical properties of the exposed specimens were generally very small and that there were no conspicuous and consistent differences among the three alloys tested. A review of the tests made on the storage specimens over a 10-yr. period (Table VI) disclosed that no really significant change in mechanical properties of the blanks had occurred; that is, the specimens were not susceptible to natural aging at room temperature. The mechanical properties of the individual specimens and the average values for the sets of specimens tested at different periods exhibited the usual variations for die castings. Furthermore, the properties of the specimens tested by the two testing stations had varied somewhat. Consequently, the average of all of these sets of storage specimens (100 specimens) represents more accurately the mean mechanical properties of each lot than do the properties determined on but one set (10 specimens) of storage specimens.

TABLE II.—INVESTIGATION OF ALUMINUM DIE-CASTING ALLOYS IVa AND Va—AVERAGE RESULTS WITHOUT REGARD TO TESTING STATION OF EXPOSURE TO THE ATMOSPHERE FOR 5 AND 10 YR.

Exposed to Atmosphere at:	Percentage Change in Tensile Strength						Percentage Change in Elongation					
	Alloy IVa		Alloy Va		Alloy V		Alloy IVa		Alloy Va		Alloy V	
	5 yr.	10 yr.	5 yr.	10 yr.	5 yr.	10 yr.	5 yr.	10 yr.	5 yr.	10 yr.	5 yr.	10 yr.
EXPOSED SPECIMENS COMPARED WITH AVERAGE OF 5 OR 10 YR. STORAGE SPECIMENS												
	(14865)		(14866)		(14867)		(14865)		(14866)		(14867)	
Sandy Hook	−2.8	−2.4	−3.2	−1.7	−1.4	−2.8	−3.4	+0.9	−10.7	+16.0	−4.3	−6.[illegible]
New York	−3.1	−5.2	−3.5[a]	−5.6	−2.9	−6.7	−3.4	−13.6	−25.0[a]	−7.3	−8.7	+0.9
Altoona	−3.0	−7.1[b]	−4.3	−3.2	−1.4	−5.7	+1.7	−6.3[b]	−3.6	−12.2	−13.0	+10.4
Average	−3.0	−4.9	−3.7	−3.5	−1.9	−5.1	−1.7	−6.3	−13.1	−1.2	−8.7	+1.[illegible]
	(15431)		(15432)		(15433)		(15431)		(15432)		(15433)	
Sandy Hook	−2.9	−3.6	−3.3	−5.5	−11.5	−8.1	−3.8	+3.7	−5.0	+3.2	−23.5	−55.0
New York	−3.7	−5.2	−10.5	−4.6	−6.2	−5.2	−7.5	−13.9	−25.0	+5.8	+5.9	−13.6
Altoona	−5.6	−4.8[b]	−5.8	−6.4	−3.0	−6.5	−11.3	+1.2[b]	−25.0	+19.6	−11.8	+10.[illegible]
Average	−4.1	−4.5	−6.5	−5.5	−6.9	−6.6	−7.5	−3.0	−18.3	+9.5	−9.5	−19.5
EXPOSED SPECIMENS COMPARED WITH AVERAGE OF ALL STORAGE SPECIMENS												
	(14865)		(14866)		(14867)		(14865)		(14866)		(14867)	
Sandy Hook	−3.2	−3.3	−3.4	−1.9	−4.8	−3.3	−5.9	−6.7	0.0	−14.1	+4.6	−26.6
New York	−3.6	−6.0	−3.8	−5.6	−6.3	−7.1	−4.1	−19.9	−17.4	−24.5	−2.3	−21.6
Altoona	−3.4	−7.9	−4.6	−3.3	−4.8	−6.1	0.0	−13.4	+1.8	−26.7	−4.6	−10.5
Average	−3.4	−5.7	−3.9	−3.6	−5.3	−5.5	−3.3	−13.3	−5.2	−21.8	−0.8	−19.6
	(15431)		(15432)		(15433)		(15431)		(15432)		(15433)	
Sandy Hook	0.0	−2.5	−1.6	−9.4	−7.9	−5.6	+4.0	−3.0	+22.7	−13.4	−8.0	−65.4
New York	−0.9	−4.3	−9.1	−8.5	−2.4	−2.6	+1.0	−20.0	−3.2	−12.9	−34.1	−19.3
Altoona	−2.9	−3.8	−4.2	−10.2	−2.4	−4.3	−3.3	−5.2	−3.2	−0.2	+8.8	−7.4
Average	−1.3	−3.5	−5.0	−9.4	−4.2	−4.2	+0.6	−9.1	+5.4	−8.8	−11.6	−30.7

[a] Average of nine specimens.
[b] Average of eight specimens.

In Table II the changes in mechanical properties of the exposed specimens tested by the two testing stations are averaged. This table contains two sets of data, one being based on a comparison with a single set of storage specimens tested concurrently with the exposed specimens, the other being based on a comparison with the average mechanical properties of all storage specimens tested over a 10-yr. period. For purposes of comparison, similar data on the 5-yr. sets of specimens are included. It is apparent from this table that the use of the average properties of all storage specimens has resulted in reducing the scatter in the test data and placing the results more in line with those obtained at the 5-yr. period.

The presentation of the data has been further simplified in Table III by averaging the changes in tensile strength for all specimens of each lot without regard to the exposure station. It is noteworthy that, on the average, the losses in tensile strength for the three alloys were very small and that the losses after 10-yr. exposure were generally slightly greater than those after 5-yr. exposure.

TABLE III.—INVESTIGATION OF ALUMINUM DIE-CASTING ALLOYS IVa AND Va—SUMMARY OF AVERAGE LOSSES IN TENSILE STRENGTH OF EXPOSED SPECIMENS WITHOUT REGARD TO EXPOSURE STATION.

Alloy	Coefficient of Variation[a] of Tensile Strength, per cent		Average Loss in Tensile Strength of Specimens Exposed to the Atmosphere for 5 or 10 yr., per cent			
	10-yr.[b] Storage Specimens	10-yr.[c] Exposed Specimens	5 yr.[d]	10 yr.[d]	5 yr.[e]	10 yr.[e]
14865 (IVa)	3.0	3.9	3.0	4.9	3.4	5.7
15431 (IVa)	3.4	5.4	4.1	4.5	1.3	3.5
Average	3.2	4.7	3.6	4.7	2.4	4.6
14866 (Va)	3.2	4.7	3.7	3.5	3.9	3.6
15432 (Va)	9.2	11.0	6.5	5.5	5.0	9.4
Average	6.2	7.9	5.1	4.5	4.5	6.5
14867 (V)	6.9	4.0	1.9	5.1	5.3	5.5
15433 (V)	8.1	10.9	6.9	6.6	4.2	4.2
Average	7.5	7.5	4.4	5.9	4.8	4.9

[a] Coefficient of variation, $v = \frac{100 \times \text{standard deviation}}{\text{mean tensile strength}}$.

[b] Ten specimens of each lot tested by two testing stations

[c] Thirty specimens of each lot tested by two testing stations.

[d] Exposed specimens compared with single set of ten storage specimens tested at corresponding 5- or 10-yr. periods.

[e] Exposed specimens compared with average of all sets of storage specimens (about 100) tested over a 10-yr. period.

A statistical analysis[4] of the data relative to the 10-yr. blanks and the 10-yr. exposure specimens was made. The coefficients of variation of these specimens are included in this table. The studies disclosed that the average losses in tensile strength for the 10-yr. exposure specimens in Table III were definitely significant, but that no significant differences existed among the three different alloys or between lots of the same alloy cast by two producers.

TABLE IV.—INVESTIGATION OF ALUMINUM DIE-CASTING ALLOYS IVa AND Va—AVERAGE PERCENTAGE LOSS IN TENSILE STRENGTH, WITHOUT REGARD TO ALLOYS, OF SPECIMENS EXPOSED TO THE ATMOSPHERE AT THE THREE STATIONS FOR 5 OR 10 YR.

Enviroment	5 yr.[a]	10 yr.[a]	5 yr.[b]	10 yr.[b]
Sandy Hook, N. J.	4.2	4.0	3.5	4.3
New York, N. Y.	5.0	5.4	4.4	5.7
Altoona, Pa.	3.9	5.6	3.7	5.9
Average	4.4	5.0	3.9	5.3

[a] Exposed specimens compared with single set of storage specimens (10) tested at corresponding 5- or 10-yr. periods.

[b] Exposed specimens compared with average of all sets of storage specimens (100) tested over a period of 10 yr.

The losses in tensile strength for all alloys are summarized in Table IV in order to express quantitatively the relative corrosiveness of the three atmospheric stations for these alloys. A statistical analysis of these data showed that the average losses in tensile strengths for specimens exposed at each station were significant, but that the differences among the three stations were not really significant. The corrosiveness of these three stations was of about the same order of magnitude.

Visual examination of the flat specimens disclosed that all specimens were in a good state of preservation and only mildly corroded. Specimens that had been exposed at Sandy Hook were light in color and contained films of white corrosion product; those that had been exposed at New York were moderately darkened, the surfaces that faced skyward being uniform in appearance and the under surfaces being speckled with spots of white corrosion product. The top surfaces of the specimens that had been exposed at Altoona were blackened with a thick film of industrial products and speckled with local spots of white product. The under surfaces were less darkened and less speckled than the top surfaces. No marked differences in surface appearance or extent of corrosive attack were evident among the three alloys or between the two lots of the same alloy cast by different producers. Specimens of alloy IVa, however, usually had maintained somewhat better surface appearance and seemed to have suffered less attack than specimens of the other two alloys. These observations were generally confirmed by depth-of-attack measurements (Table V) made on transverse sections of the exposed specimens. From these data it is noteworthy that the rate of attack is definitely decelerating with time. The depth of attack after 10-yr. exposure was, on the average, not very much greater than that measured after 5-yr. exposure.

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[4] R. A. Fisher, "Statistical Methods for Research Workers," Oliver and Boyd, Edinburgh, p. 128.

TABLE V.—RESULTS OF MICROSCOPIC EXAMINATION OF FLAT SPECIMENS EXPOSED FOR EITHER 5 OR 10 YR.[a]

Alloy	Depth of Attack, mils											
	Sandy Hook, N. J.				New York, N. Y.				Altoona, Pa.			
	Maximum		Average		Maximum		Average		Maximum		Average	
	5 yr.	10 yr.	5 yr.	10 yr.	5 yr.	10 yr.	5 yr.	10 yr.	5 yr.	10 yr.	5 yr.	10 yr.
No. 14865 (IVa)	6.7	5.3	3.6	3.6	4.2	9.8	3.4	5.4	6.4	8.4	5.0	5.8
No. 15431 (IVa)	4.2	4.4	3.4	3.2	4.5	6.1	3.6	4.0	7.0	missing	4.8	missing
No. 14866 (Va)	5.9	8.4	4.5	4.1	4.8	9.2	3.6	5.9	6.2	missing	4.8	missing
No. 15432 (Va)	5.6	7.0	3.9	4.6	4.5	6.7	3.4	4.0	7.0	7.0	4.5	4.4
No. 14867 (V)	6.2	5.6	4.5	4.9	6.4	8.9	4.8	5.3	5.6	7.2	4.2	5.4
No. 15433 (V)	5.6	6.1	3.6	4.4	4.5	6.7	3.9	4.6	7.3	7.2	4.8	5.2

[a] Measurements were made at a magnification of 100 on one transverse section of specimens exposed 5 yr. and on two transverse sections of specimens exposed 10 yr. All specimens were corroded principally by attack of the constituent network.

Conclusions:

The results of the tests and examination of specimens exposed for 10 yr. at Sandy Hook, New York, and Altoona show that alloys IVa, Va, and V all exhibit a high resistance to corrosion. These exposures caused, on the average, small but significant losses in the strength of the three alloys, but there were no really significant differences among the three alloys or between the two lots of the same alloy cast by different producers. These observations, based on mechanical data, were generally confirmed by visual and microscopic examinations of the specimens. One consistent difference observed visually was that specimens of alloy IVa had maintained a somewhat better appearance than did specimens of alloys Va and V.

It is noteworthy from a comparison of the 5- and 10-yr. data in Tables III and V that the corrosion of the specimens is occurring at a definitely decelerating rate. Consequently, it is likely that the losses in mechanical properties of the specimens will be of a small order of magnitude even after a

TABLE VI.—INVESTIGATION OF ALUMINUM DIE-CASTING ALLOYS NOS. IVa AND Va—SUMMARY OF MECHANICAL PROPERTIES OF STORAGE SPECIMENS TESTED AS BLANKS WITH VARIOUS CORROSION PERIODS.[a]

Date Tested	Approximate Storage Period	Tensile Strength, psi.						Yield Strength, psi.						Elongation in 2 in., per cent						Tested by[b]	Number Tested
		Alloy IVa		Alloy Va		Alloy V		Alloy IVa		Alloy Va		Alloy V		Alloy IVa		Alloy Va		Alloy V			
		14865	15431	14866	15432	14867	15433	14865	15431	14866	15432	14867	15433	14865	15431	14866	15432	14867	15433		
September 4, 1934	3 months	26 445	25 600	32 200	27 555	34 630	28 365	9 850	11 950	16 450	17 325	18 575	18 850	5.7	4.8	2.3	1.2	2.0	1.1	ARL	10
January 25, 1935	7 months	27 340	25 820	32 815	28 950	35 818	29 155	10 025	12 450	16 575	18 100	19 000	19 325	6.1	5.2	2.4	1.8	2.3	1.4	ARL	10
May 6, 1935	11 months	26 955	26 120	33 490	27 655	36 715	28 245	10 100	12 450	16 600	17 300	18 700	18 700	5.9	5.0	2.9	1.4	2.3	1.4	ARL	5
July 2, 1935	13 months	27 390	25 960	32 825	25 765	36 010	28 345	9 750	12 200	16 600	16 800	18 950	19 400	6.7	4.8	2.3	1.5	2.3	1.4	ARL	5
October 4, 1935	16 months	27 820	26 800	33 945	28 630	37 180	27 965	10 350	12 800	16 950	16 850	19 500	19 150	6.4	5.1	2.5	1.8	2.4	1.3	ARL	5
November 5, 1935	17 months	27 820	25 740	33 710	28 870	37 400	30 380	10 250	12 350	16 800	17 900	19 500	19 600	6.3	4.7	2.3	1.6	2.2	1.7	ARL	5
January 4, 1940	5½ yr.	27 425	26 540	33 530	29 820	35 370	30 140	10 500	12 650	16 600	17 500	19 500	20 350	6.3	5.2	3.0	2.3	2.5	1.8	ARL	5
January 4, 1945	10½ yr.	26 700	25 900	34 260	26 000	33 980	30 760	10 740	12 880	16 960	16 680	20 460	20 640	5.5	4.8	2.7	1.5	1.9	1.3	ARL	5
Average		27 237	26 060	33 347	27 906	36 138	29 169	10 196	12 466	16 692	17 307	19 273	19 502	6.1	5.0	2.6	1.6	2.2	1.4		
September 15, 1934	3 months	28 260	25 140	32 420	30 900	38 080	29 060							6.1^e	4.8^d	2.1^e	2.0	2.4^e	1.5^e	WEC	5
January 31, 1935	7 months	28 500	27 120	35 400	29 445	39 005	29 860							4.9	3.8	2.2	1.4	1.7	1.0	BTL	10
May 1935	11 months	29 100	27 500	35 650	28 760	38 620	30 260							5.8	4.7	2.2	1.3	1.6	1.5	BTL	5
July 31, 1935	14 months	27 800	25 680	33 480	29 340	38 560	27 620							6.8^e	4.8^e	2.8^c	1.6^e	2.3^c	1.5^d	WEC	5
November 6, 1935	17 months	28 660	26 020	34 940	29 240	38 380	30 800							6.2^d	4.7	2.4^d	1.3^c	2.0^d	1.1^e	WEC	5
November 15, 1935	17 months	28 510	26 670	37 380	29 710	36 960	31 490							6.3	4.9	2.8	1.6	2.8	1.5	BTL	10
January 3, 1940	5½ yr.	27 980	27 700	34 280	28 420	36 160	31 520							5.5	5.3	2.5^e	1.7	2.0	1.5	WEC	5
February 25, 1945	10½ yr.	28 482	27 376	$33\,695^e$	28 990	37 756	29 984							5.6	4.3	1.5^e	1.1	1.4	1.0	WEC	5
Average		28 412	26 651	34 656	29 351	37 940	30 074							5.9	4.7	2.4	1.5	2.0	1.3		

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[a] Alloys Nos. 14865, 14866, 14867, (Nos. IVA, Va, V, respectively) were cast by the Aluminum Company of America on June 6, 5, 7, 1934, respectively. Alloys Nos. 15431, 15432, 15433, (Nos. IVa, Va, V, respectively) were cast by the General Electric Co. on June 28, 25, 16, 1934, respectively.

[b] ARL = Aluminum Research Laboratories, BTL = Bell Telephone Laboratories, WEC = Western Electric Co., Hawthorne Plant.

[c] Average of two specimens.

[d] Average of three specimens.

[e] Average of four specimens.

considerably longer period of exposure, and that no really marked differences are to be expected among the three alloys under study. Thus it would seem that the superior rating given alloy Va in the accelerated tests has not yet been confirmed by the atmospheric exposures. The accelerated and atmospheric tests to date indicate that small differences in the inherent resistance to corrosion of the three alloys might exist if they are exposed to severely corrosive conditions, such as are typified by the salt spray. Under the less corrosive conditions of exposure most likely to be encountered in service, little difference among the three alloys is to be expected.

Respectfully submitted on behalf of the subcommittee,

J. J. BOWMAN,
Chairman

REPORT OF SUBCOMMITTEE III ON TIN- AND LEAD-BASE DIE CASTINGS

This report presents the results of creep tests in tension obtained in furtherance of the investigation of the mechanical properties of tin- and lead-base alloy die-cast specimens which has been undertaken by Subcommittee III. This investigation was accomplished through the cooperation of two die casters who supplied the test specimens and four other companies who supplied the ingot metal. The creep tests were made by the Bell Telephone Laboratories, Inc.

TABLE I.—SPECIFIED REQUIREMENTS FOR CHEMICAL COMPOSITIONS OF LEAD- AND TIN-BASE ALLOYS.

Alloy	Tin, per cent			Antimony, per cent			Lead, per cent			Copper, per cent			Iron, max. per cent	Arsenic, max. per cent	Zinc, max. per cent	Aluminum, max. per cent
	Min.	Desired	Max.	Min.	Desired	Max.	Min.	Desired	Max.	Min.	Desired	Max.				
No. 1	90	91	92	4	4.5	5	...	...	0.35	4	4.5	5	0.08	0.08	0.01	0.01
No. 2	80	82	84	12	13	14	...	...	0.35	4	5	6	0.08	0.08	0.01	0.01
No. 3	64	65	66	14	15	16	17	18	19	1.5	2	2.5	0.08	0.15	0.01	0.01
No. 4	4	5	6	14	15	16	79	80	81	...	...	0.50	...	0.15	0.01	0.01
No. 5	...	...	...	9.25	10	10.75	89	90	91	...	...	0.50	...	0.15	0.01	0.01

Flat and round die-cast specimens were used in conducting the tests. Chemical analyses, tensile strength and elongation data pertaining to the same lots of specimens have been previously reported.[1] Reference thereto, as well as to the reprinted data shown in Tables I and II, disclose the fact that the round specimens of alloys Nos. 1 and 2 are definitely lower in copper content than the limit specified, in A.S.T.M. Standard Specifications for Lead- and Tin-Base Alloy Die Castings (B 102 – 44).[2]

The creep tests were made of both flat and round tension specimens of each of the five tin- and lead-base die casting alloys using a 2-in. gage length. The technique described elsewhere[3] was used in these studies. Dead-weight loads were applied to the specimens to develop the 1000 psi. stress; for higher stresses a lever system was used. Extension of the specimens was measured periodically by means of a cathetometer reading to 0.002 in., although estimation to the nearest 0.001 in. could be made. On this basis the elongation measurements may be considered to have a precision of ±0.1 per cent.

The elongation-time data are given in Tables III to VII inclusive and in Figs. 1 to 5. A summary of the stress-rupture data is given in Table VIII. The unbroken creep specimens, which were removed on April 17, 1946, when the tests were discontinued, were broken in the standard tension test to determine the effect of long sustained creep loads on the tensile properties. The results

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[1] Report of Subcommittee III on Tin- and Lead-Base Die Casting Alloys, Report of Committee B-6, *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 282 (1939).

[2] 1944 Book of A.S.T.M. Standards, Part I, p. 884.

[3] G. R. Gohn, S. M. Arnold, and G. M. Bouton, "Creep Tests on Some Extruded Lead and Lead-Alloy Sleeves and Tapes," see p. 990.

TABLE II.—CHEMICAL ANALYSES OF FRACTURED TENSION SPECIMENS.

Alloy	Specimens	Analysts	Tin, per cent	Antimony, per cent	Lead, per cent	Copper, per cent	Iron, per cent	Arsenic, per cent	Zinc, per cent	Aluminum, per cent
No. 1	Flat	A	90.91	4.76	0.12	4.04	0.04	0.04	<0.01	0.001
	Flat	B	90.44	4.5	0.14	4.34	...	0.04	...	...
	Round	A	92.42	4.84	0.29	2.26	0.03	0.06	<0.01	0.001
	Round	B	92.2	5.0	0.14	2.45	...	0.05	...	...
No. 2	Flat	A	81.81	12.94	0.47	4.62	0.06	0.01	<0.01	0.001
	Flat	B	81.48	12.9	0.13	4.84	...	0.04	...	...
	Round	A	84.02	12.98	0.12	2.76	0.02	0.008	<0.01	0.001
	Round	B	82.8	13.2	0.10	3.0	...	0.05	...	...
No. 3	Flat	A	66.06	15.05	16.68	2.00	0.05	0.055	<0.01	0.001
	Flat	B	65.40	15.0	17.02	2.20	...	0.04	...	...
	Round	A	66.11	14.27	18.01	1.42	0.02	0.07	<0.01	0.001
	Round	B	66.0	14.1	17.8	1.70	...	0.05	...	0.001
No. 4	Flat	A	4.81	14.88	80.13	0.02	0.02	0.09	<0.01	0.001
	Flat	B	4.70	15.4	79.6	0.14	...	0.06	...	...
	Round	A	4.93	14.60	80.20	0.14	0.02	0.085	<0.01	0.001
	Round	B	5.0	14.6	79.80	0.30	...	0.07	...	...
No. 5	Flat	A	0.21	8.88	90.73	0.04	0.01	0.04	<0.01	0.001
	Flat	B	...	8.8	90.6	0.10	...	0.04	...	...
	Round	A	0.06	9.85	89.90	0.09	0.02	0.06	<0.01	0.001
	Round	B	...	9.8	89.8	0.20	...	0.05	...	...

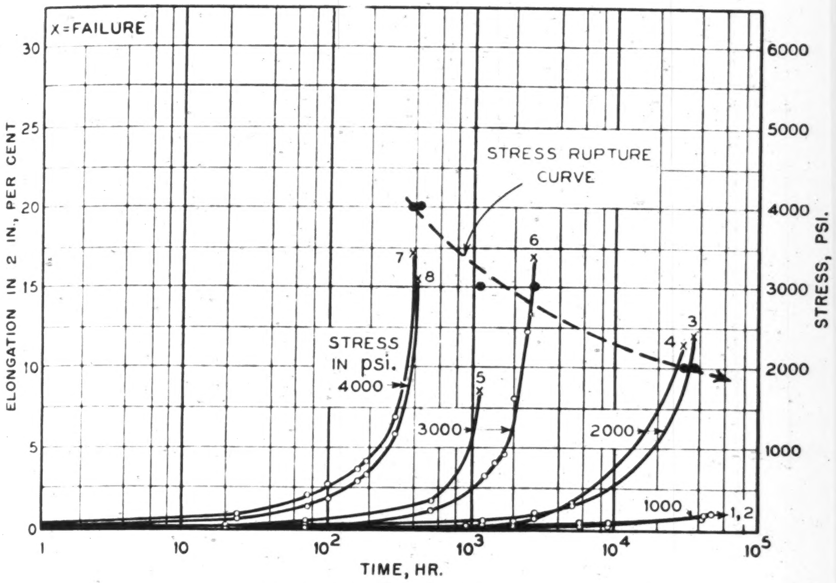
TABLE III.—CREEP DATA ON TIN-BASE DIE-CASTING ALLOY NO. 1.

Nominal Composition, Per cent

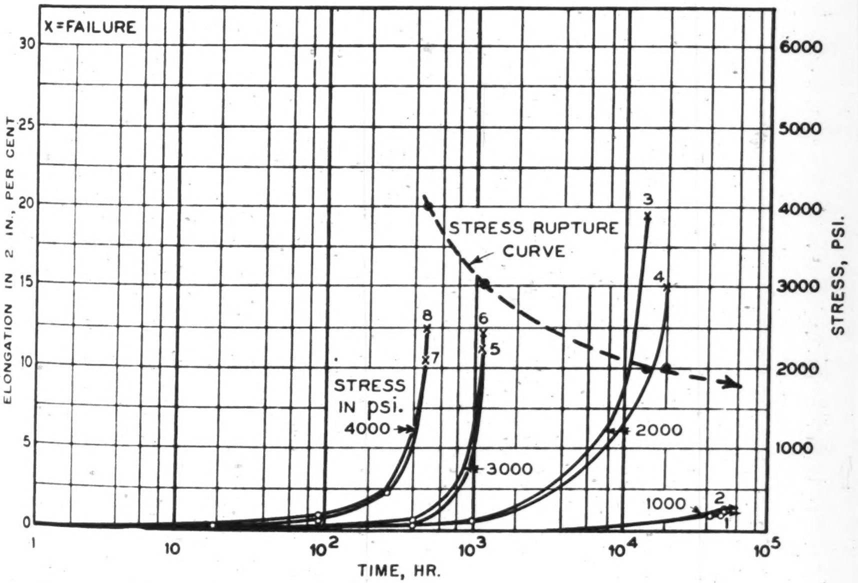
Tin	91.0
Antimony	4.5
Copper	4.5[a]

Initial Stress, psi	4000				3000				2000				1000			
Specimen No.	1-7		1-8		1-5		1-6		1-3		1-4		1-1		1-2	
	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent
FLAT SPECIMENS																
	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0
	[b]	0.10	[b]	0.05	[b]	0.20	[b]	0.10	[b]	0.15	[b]	0.0	[b]	0.0	[b]	0.05
	24	0.85	24	0.55	20	0.35	20	0.10	72	0.30	72	0.0	916	0.15	916	0.05
	76	1.95	76	1.35	72	0.45	72	0.10	1 245	0.55	1 245	0.35	2 088	0.15	2 088	0.10
	101	2.60	101	1.85	528	1.75	528	1.00	2 016	0.55	2 016	0.45	5 784	0.35	5 784	0.20
	168	3.60	168	2.85	1171	8.6[c]	1248	3.20	2 712	0.95	2 712	0.55	9 192	0.35	9 192	0.20
	195	4.10	195	3.20			1440	4.00	4 944	1.35	4 944	1.40	42 216	0.60	42 216	0.60
	288	6.7	288	5.8			1608	4.60	34 144	12.7[c]	29 932	11.3[c]	43 512	0.70	43 512	0.70
	384	17.0[c]	413	15.6[c]			2040	8.0					48 053	0.70	48 053	0.70
							2448	12.3					49 609[d]	0.70[d]	49 609[d]	0.70[d]
							2616	18.5[c]								
ROUND SPECIMENS[a]																
	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0
	[b]	0.05	[b]	0.05	[b]	0.0	[b]	0.0	984	0.50	984	0.40	[b]	0.0	[b]	0.0
	19	0.10	19	0.20	139	0.20	139	0.55	14 760	19.5[c]	19 632	15.0[c]	912	0.0	912	0.0
	91	0.65	91	0.80	1157	11.0[c]	1891	12.0[c]					1 368	0.0	1 368	0.0
	259	2.20	259	2.30									5 088	0.0	5 088	0.0
	435	10.5[c]	435	12.5[c]									42 384	0.80	42 384	0.90
													48 053	0.80	48 053	1.10
													49 609[d]	1.00[d]	49 609[d]	1.20[d]

[a] The copper content of the round tension bars was found to be only 2.3 per cent.
[b] Denotes initial elongation upon application of load.
[c] Denotes total elongation after fracture of test specimen.
[d] No failure, test discontinued on April 17, 1946.



(*a*) Flat Tension Specimens.



(*b*) Round Tension Specimens (copper content was found to be only 2.3 per cent).

FIG. 1.—Creep of Tin-Base Die Casting Alloy No. 1.—91 per cent Tin, 4.5 per cent Antimony, and 4.5 per cent Copper.

TABLE IV.—CREEP DATA ON TIN-BASE DIE-CASTING ALLOY NO. 2.

Nominal Composition, per cent

Tin, 82.0; antimony, 13.0; copper, 5.0[a]

Initial Stress, psi......	4000				3000				2000				1000			
Specimen No......	2-7		2-8		2-5		2-6		2-3		2-4		2-1		2-2	
	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation
FLAT SPECIMENS																
	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0
	[b]	0.0	[b]	0.0	[b]	0.10	[b]	0.10	[b]	0.10	[b]	0.10	[b]	0.0	[b]	0.0
	24	0.50	24	0.45	20	0.15	20	0.30	72	0.10	72	0.20	5 784	0.20	5 784	0.1
	76	1.05	76	0.95	72	0.20	72	0.40	1 245	0.25	1 245	0.80	42 216	0.60	42 216	0.6
	101	1.25	101	0.95	528	0.80	528	1.30	2 016	0.35	2 016	0.80	43 512	0.60	43 512	0.6
	168	1.70	168	1.30	1248	1.30	1248	2.50	2 712	0.55	2 712	1.10	48 053	0.70	48 053	0.6
	288	2.75	288	2.10	1440	1.80	1440	2.70	4 944	0.60	4 944	1.15	49 609[d]	0.70[d]	49 609[d]	0.6
	384	4.00	384	2.90	2040	2.40	2040	4.60	41 596	3.25	41 596	5.3				
	504	5.8	504	4.70	2448	3.30	2448	5.6	42 792	3.65	42 732	5.6				
	699	12.0[c]	533	7.0[c]	2712	3.30	2712	7.4	47 133	3.95	47 133	6.6				
					3096	3.60	2760	9.0[c]	48 689[d]	4.25[d]	48 689[d]	6.6[d]				
					5016	7.1										
					5784	12.0[c]										
ROUND SPECIMENS[a]																
	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0
	[b]	0.20	[b]	0.05	[b]	0.0	[b]	0.0	984	0.55	984	0.10	[b]	0.0	[b]	0.0
	19	0.30	19	0.15	139	0.40	139	0.30	26 400	23.0[c]	35 986	54.4[c]	912	0.0	912	0.0
	91	0.50	91	0.30	1171	2.35	1171	2.75					5 088	0.0	5 088	0.0
	259	2.90	259	1.50	2131	8.2	2131	8.3					42 384	0.65	42 384	0.6
	504	8.4	504	9.0	2736	18.1[c]	2976	32.0[c]					48 053	0.65	48 053	1.0
	624	19.0	624	13.3									49 609[d]	0.75[d]	49 609[d]	1.1
	650	25.0[c]	666	16.5[c]												

[a] The copper content of the round tension bars was found to be only 2.9 per cent.
[b] Denotes initial elongation upon application of load.
[c] Denotes total elongation after fracture of test specimen.
[d] No failure, test discontinued on April 17, 1946.

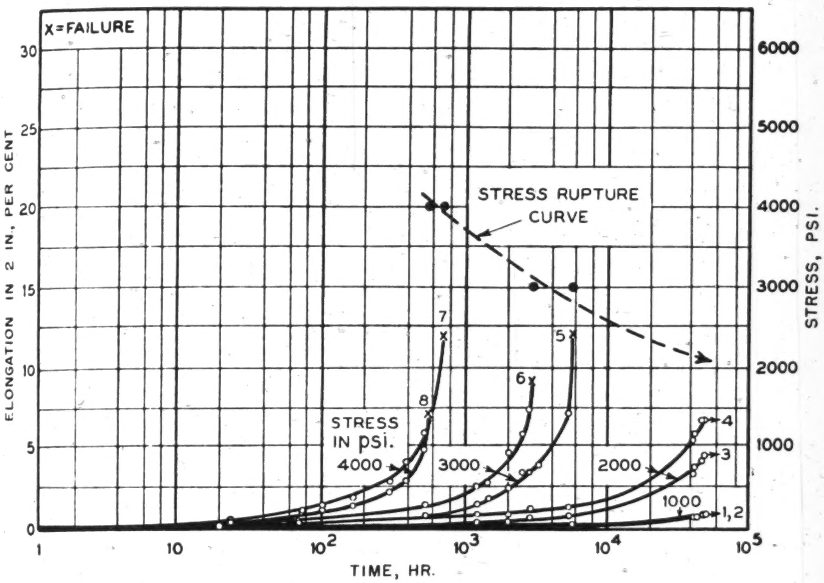
TABLE V.—CREEP DATA ON TIN-BASE DIE-CASTING ALLOY NO. 3.

Nominal Composition, per cent

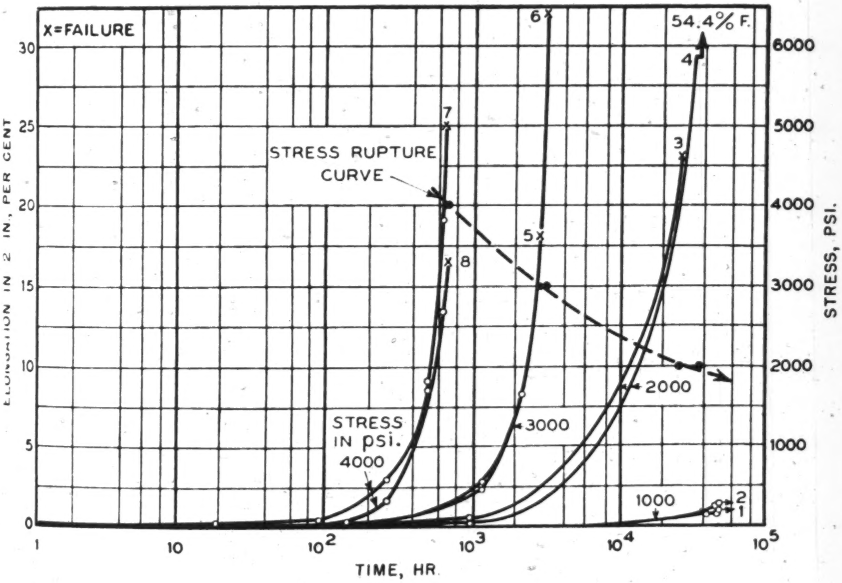
Tin, 65.0; antimony, 15.0; lead, 18.0; copper, 2.0

Initial Stress, psi...........	3000				2000				1000			
Specimen No...	3-5		3-6		3-7		3-8		3-1		3-2	
	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent
FLAT SPECIMENS												
	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0
	[a]	0.15	[a]	0.10	[a]	0.20	[a]	0.10	[a]	0.20	[a]	0.0
	20	0.15	20	0.55	72	0.25	72	0.10	916	0.35	916	0.05
	72	1.40	72	1.70	168	0.45	168	0.10	2 088	0.40	2 088	0.15
	99	2.15	99	2.85[b]	500	0.75	500	0.60	5 784	0.65	5 784	0.35
	139	3.80[b]			936	2.50[b]	936	2.10	9 192	2.50[b]	42 216	1.25
							1158	3.20[b]			43 512	1.55
											48 837	1.60
ROUND SPECIMENS												
	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0
	[a]	0.15	[a]	0.10	960	9.0[b]	984	2.50	[a]	0.0	[a]	0.0
	48	2.45	48	3.20			1080	11.5[b]	912	0.0	5 088	0.15
	72	3.70	72	6.5					1 368	0.15	22 584	6.5
	127	10.0[b]	86	9.5[b]					5 088	0.65		
									25 108	9.0[b]		

[a] Denotes initial elongation upon application of load.
[b] Denotes total elongation after fracture of test specimen.
[c] Broke outside of gage marks



(*a*) Flat Tension Specimens



(*b*) Round Tension Specimens (copper content was found to be only 2.9 per cent).

Fig. 2.—Creep of Tin-Base Die Casting Alloy No. 2.—82 per cent Tin, 13 per cent Antimony, and 5.0 per cent Copper.

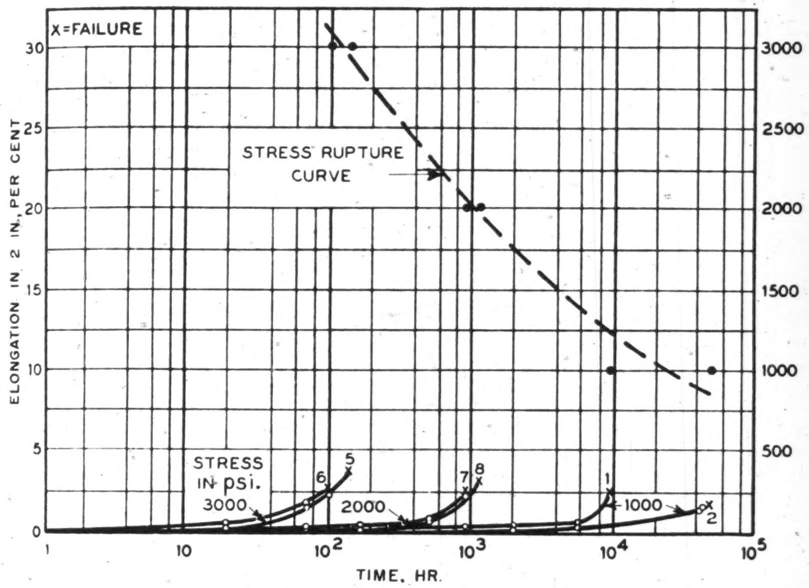
TABLE VI.—CREEP DATA ON LEAD-BASE DIE-CASTING ALLOY NO. 4.

Nominal Composition, per cent

Tin	5.0
Antimony	15.0
Lead	80.0

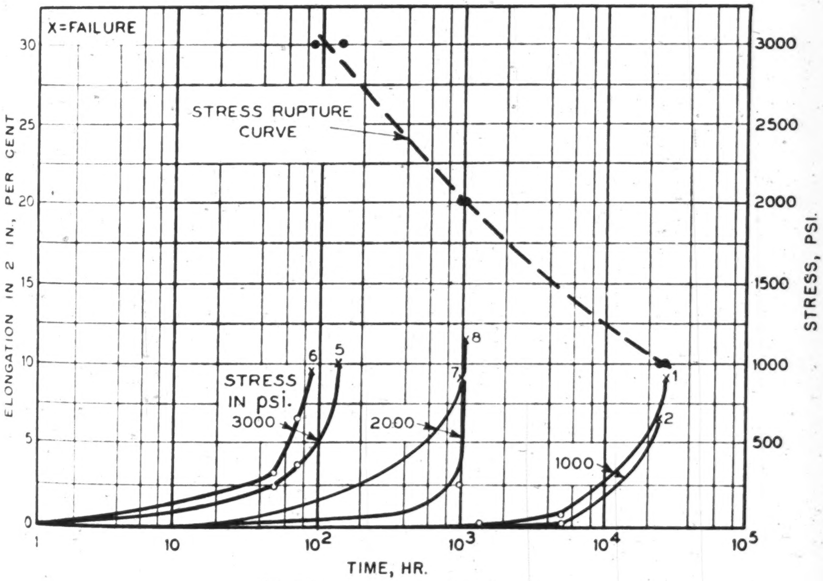
Initial Stress, psi	4000				3000				2000				1000			
Specimen No.	4-7		4-8		4-5		4-6		4-3		4-4		4-1		4-2	
	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent
FLAT SPECIMENS																
	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0
	[a]	0.05	[a]	0.0	[a]	0.10	[a]	0.05	[a]	0.0	[a]	0.10	[a]	0.0	[a]	0.0
	24	0.40	24	0.40	20	0.15	20	0.15	72	0.30	72	0.10	916	0.0	916	0.0
	76	0.70	76	0.65	72	0.30	72	0.20	1 245	0.40	1 245	0.10	5 784	0.0	5 784	0.0
	101	1.25	101	0.90	528	0.50	528	0.40	2 016	0.40	2 016	0.10	42 216	0.0	42 216	0.1
	168	1.25	168	0.95	1248	0.50	1 248	0.45	2 712	0.50	2 712	0.15	43 516	0.0	43 512	0.1
	288	1.25	288	1.00	2040	0.60	2 040	0.55	4 944	0.50	4 944	0.20	48 053	0.10	48 053	0.1
	384	1.45	384	1.45	2712	0.70	2 712	0.86	41 596	1.00	41 596	0.60	49 609[e]	0.10[e]	49 609[e]	0.1
	504	1.95	504	1.70	3096	0.70	3 096	0.90	42 796	1.00	42 792	0.60				
	555	3.70[b, c]	548	2.00[b, c]	5016	1.05	5 016	1.05	47 133	1.00	47 133	0.60				
					5886	1.15	5 760	2.00[b, c]	48 689[e]	1.00[e]	48 689[e]	0.60[e]				
					7536	3.50[b]										
ROUND SPECIMENS																
	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0				
	[a]	0.10	[a]	0.25	[a]	0.05	[a]	0.15	984	0.15	984	0.0	0	0.0	0	0.0
	19	0.20	19	0.35	103	0.05	103	0.15	...	...	1 824	0.20	912	0.0	912	0.0
	91	0.40	91	0.50	139	0.20	139	0.40	35 760	1.05	35 762	0.55	1 368	0.0	1 368	0.0
	259	0.70	259	0.85	1891	0.60	1 891	0.70	38 712	1.05	38 712	0.55	3 216	0.0	3 216	0.0
	504	1.80	504	3.00[b, d]	2131	1.10	2 131	0.95	43 253	1.05	43 253	0.55	5 088	0.0	5 088	0.2
	624	2.00			4252	4.50[b]	12 960	3.00[b]	44 809[e]	1.25[e]	44 809[e]	0.55[e]	42 384	0.30	42 384	0.4
	710	3.50											48 053	0.30	48 053	0.4
	853	5.0[b]											49 609[e]	0.50[e]	49 609[e]	0.4

[a] Denotes initial elongation upon application of load.
[b] Denotes total elongation after fracture of test specimen.
[c] Broke outside of gage marks.
[d] Broke in gage mark.
[e] No failure, test discontinued on April 17, 1946.



(*a*) Flat Tension Specimens.

Note.—Specimen No. 2 broke outside of gage marks.



(*b*) Round Tension Specimens.

Fig. 3.—Creep of Tin-Base Die Casting Alloy No. 3.—65.0 per cent Tin, 15.0 per cent Antimony, 18.0 per cent Lead, and 2.0 per cent Copper.

TABLE VII.—CREEP DATA ON LEAD-BASE DIE-CASTING ALLOY NO. 5.

Nominal Composition, per cent

Antimony	10.0
Lead	90.0

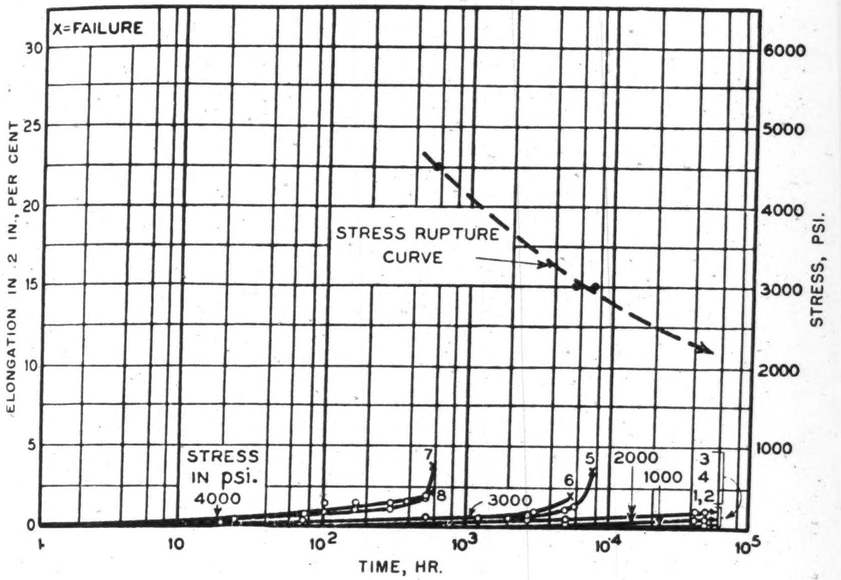
Initial Stress, psi.	3000				2000				1000			
Specimen No.	5-3		5-4		5-7		5-8		5-1		5-2	
	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent	Time, hr.	Elongation in 2 in., per cent
FLAT SPECIMENS												
	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0	0	0.0
	[a]	0.20	[a]	0.0	[a]	0.10	[a]	0.05	[a]	0.0	[a]	0.0
	20	1.00	20	0.40	72	0.20	72	0.20	916	0.05	916	0.15
	72	1.20	72	1.35	936	0.50	936	0.60	2 088	0.05	2 088	0.15
	168	2.00	168	2.10	1245	0.60	1245	0.60	5 784	0.15	5 784	0.25
	288	4.10	288	3.30	2016	1.30	2016	0.85	42 216	0.15	42 216	0.60
	360	4.30	360	4.40	2712	1.50	2712	0.85	43 512	0.40	43 512	0.60
	435	7.0[b]	384	4.60[b, c]	3384	3.20[b, c]	4536	[d]	48 053	0.40	48 053	0.60
									49 609[e]	0.40[e]	49 609[e]	0.60
ROUND SPECIMENS												
	0	0.0	0	0.0	0	0.0	0	0.0				
	103	11.4[b]	48	4.35	984	4.35	984	1.10	0	0.0	0	0.0
			72	6.0	1344	8.0[b]	1176	8.0[b, c]	3 216	0.0	3 216	0.0
			170	16.5[b]					5 088	0.45	5 088	0.8
									22 128	[d]	26 328	6.5[b]

[a] Denotes initial elongation upon application of load.
[b] Denotes total elongation after fracture of test specimen.
[c] Broke outside of gage marks.
[d] Elongation at failure not recorded.
[e] No failure, test discontinued on April 17, 1946.

TABLE VIII.—SUMMARY OF STRESS-RUPTURE DATA ON LEAD-AND TIN-BASE DIE-CASTING ALLOY

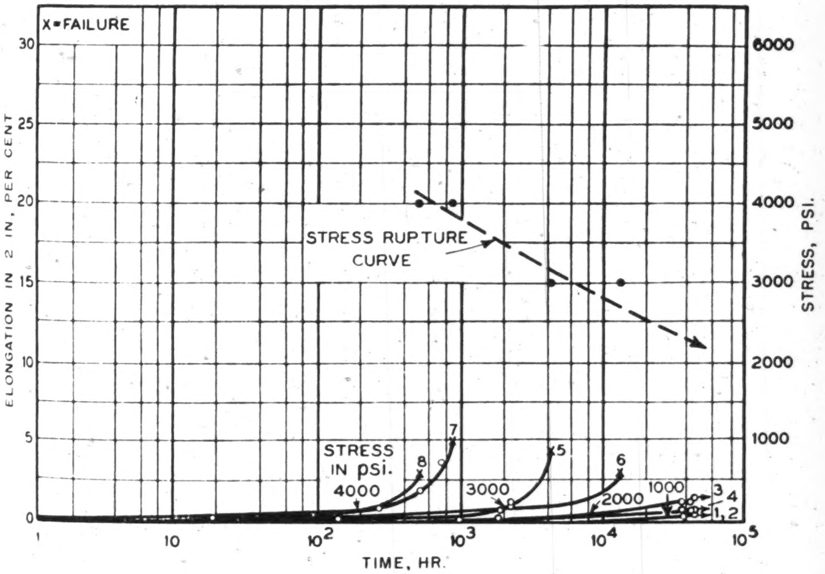
Alloy No.	Shape	Rupture Stress							
		4000 psi.		3000 psi.		2000 psi.		1000 psi.	
		Time to Failure, hr.	Elongation in 2 in. at Failure, per cent	Time to Failure, hr.	Elongation in 2 in. at Failure, per cent	Time to Failure, hr.	Elongation in 2 in. at Failure, per cent	Time to Failure, hr.	Elongation in 2 in. at Failure, per cent
1	Flat	384	17.0	1 171	8.6	34 144	12.7	49 609[a]	0.70[a]
	Flat	413	15.6	2 616	18.5	29 932	11.3	49 609[a]	0.70[a]
	Round	435	10.5	1 157	11.0	14 760	19.5	49 609[a]	1.00[a]
	Round	435	12.5	1 891	12.0	19 632	15.0	49 609[a]	1.20[a]
2	Flat	699	12.0	5 784	12.0	48 689[a]	4.25[a]	49 609[a]	0.70[a]
	Flat	533	7.0	2 760	9.0	48 689[a]	6.60[a]	49 609[a]	0.65[a]
	Round	650	25.0	2 736	18.1	26 400	23.0	49 609[a]	0.75[a]
	Round	666	16.5	2 976	32.0	35 986	54.4	49 609[a]	1.15[a]
3	Flat	...	...	139	3.80	936	2.50	9 192	3.50
	Flat	...	...	99	2.85	1 158	3.20	48 837	1.60[b]
	Round	...	...	127	10.0	960	9.0	25 108	9.0
	Round	...	...	86	9.5	1 080	11.5	22 584	6.5
4	Flat	555	3.70	7 536	3.50	48 689[a]	1.00[a]	49 609[a]	0.10[a]
	Flat	548	2.00	5 760	2.00	48 689[a]	0.60[a]	49 609[a]	0.15[a]
	Round	853	5.0	4 252	4.50	44 809[a]	1.25[a]	49 609[a]	0.50[a]
	Round	504	3.0	12 960	3.00	44 809[a]	0.55[a]	49 609[a]	0.40[a]
5	Flat	...	...	435	7.0	3 384	3.20[b]	49 609[a]	0.40[a]
	Flat	...	...	384	4.60[b]	4 536	>0.85[c]	49 609[a]	0.60[a]
	Round	...	...	103	11.4	1 344	8.0	22 128	>0.45[c]
	Round	...	...	170	16.5	1 176	8.0[b]	26 328	6.5

[a] No failure, test discontinued on April 17, 1946.
[b] Broke outside of gage marks.
[c] Elongation at failure not recorded.



(*a*) Flat Tension Specimens

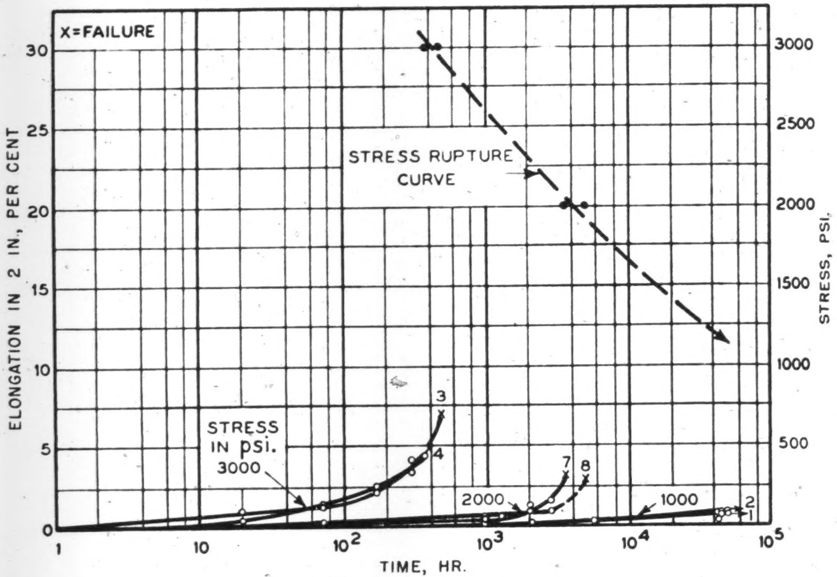
NOTE.—Specimens Nos. 6, 7, and 8 broke outside of gage marks.



(*b*) Round Tension Specimens

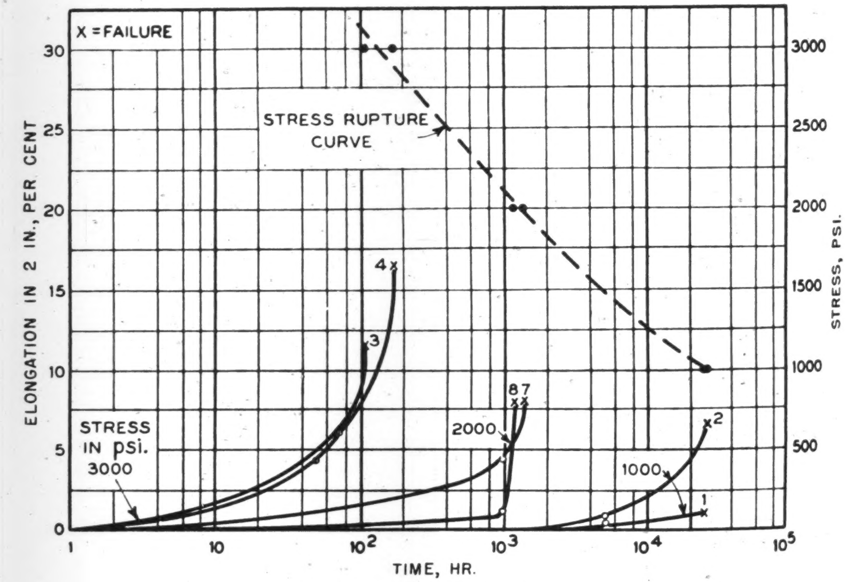
NOTE.—Specimen No. 8 broke in gage mark.

FIG. 4.—Creep of Lead-Base Die Casting Alloy No. 4.—5.0 per cent Tin, 15.0 per cent Antimony, and 80 per cent Lead.



(*a*) Flat Tension Specimens

NOTE.—Specimens Nos. 4 and 7 broke outside of gage marks. Elongation of failure not recorded for Specimen No. 8.



(*b*) Round Tension Specimens

NOTE.—Specimen No. 8 broke outside of gage marks. Elongation of failure not recorded for specimen No. 1.

FIG. 5.—Creep of Lead-Base Die Casting Alloy No. 5.—10 per cent Antimony, 90.0 per cent Lead.

TABLE IX.—SUMMARY OF TENSION TESTS ON UNBROKEN CREEP SPECIMENS.

Alloy	Sample	Tensile Strength, psi.	Elongation in 2 in., per cent
FLAT TENSION SPECIMENS			
No. 1	No. 1	8 500	5.0
	No. 2	8 800	14.5
		(9 640)[b]	(15.9)[b]
No. 2	No. 1	11 100	2.5
	No. 2	11 400	3.0
	No. 3	10 900	3.0
	No. 4	9 800	2.5
		(13 150)	(6.3)
No. 4	No. 1	9 800	6.0
	No. 2	8 800	6.0
	No. 3	8 500	7.0
	No. 4	8 500	6.0
		(10 090)	(3.7)
No. 5	No. 1	7 100	8.5
	No. 2	7 300	7.0
		(7 880)	(5.5)
ROUND TENSION SPECIMENS			
No. 1	No. 1	9 000	15.0
	No. 2	8 600	20.0
		(10 310)	(22.9)
No. 2	No. 1	10 500	8.5
	No. 2	11 100	12.5
		(13 470)	(13.2)
No. 4	No. 1	8 900	[a]
	No. 2	9 200	5.0
	No. 3	7 600	1.5
	No. 4	9 300	15.0
		(10 720)	(6.1)

[a] Broke outside gage marks. Specimens Nos. 1 and 2 were subjected to a creep stress of 1000 psi. for a period of 5⅔ yr., specimens Nos. 3 and 4 to a creep stress of 2000 psi. for a period of 5⅔ yr. prior to making the tension tests.

[b] Values in parentheses are initial values.

of these tests are given in Table IX. Comparison of these tension test data with the original tension data¹ shown in the same table indicates that the creep stress had but little effect on the tensile properties.

From these creep studies, at the loads employed, it would appear that there is definite creep in all five of the alloys after approximately 50,000 hr. Stress-rupture occurred in the flat and round specimens of alloy No. 3 and the round specimens of alloy No. 5 in 9,000 to 49,000 hr. Specimens of alloy No. 4 showed the least creep during the test period while alloys Nos. 1 and 2 had approximately the same extension at the conclusion of the tests.

Respectfully submitted on behalf of the subcommittee,

G. O. HIERS,
Chairman.

DISCUSSION

MR. HIERS.—The Bell Telephone Laboratories have presented valuable creep data. The accompanying Table X, which may be used at an engineer's own risk compares four sets of values:

1. Tensile strength data reported in 1939 and averaged.
2. One-year life values taken from the stress-rupture curves.
3 and 4. 10 year and infinite life values estimated by extra-polation from the stress-rupture curves.

TABLE X.—SHORT- AND LONG-TIME TENSILE STRENGTHS IN POUNDS PER SQUARE INCH.

Alloy	Initial		1-yr. Life		10-yr. Life		Infinite Life	
	Flat	Round	Flat	Round	Flat	Round	Flat	Round
No. 1	9 640	10 310	2300	2100	1800	1750	1500	1500
No. 2	13 150	13 470	2600	2400	2000	1750	1500	1200
No. 3	10 130	11 800	1250	1300	800	750	500	600
No. 4	10 090	10 720	2800	2800	2100	2000	1700	1700
No. 5	7 880	7 890	1700	1300	900	700	600	500

REPORT OF SUBCOMMITTEE V ON EXPOSURE AND CORROSION TESTS

This report covers the mechanical properties on: (*1*) the last of the aluminum-base and the zinc-base specimens from the exposure tests started in 1929[1] and (*2*) the first aging data on the magnesium-base and the zinc-base alloys from exposures started in 1939.[2]

Aluminum and Zinc-Base Alloys:

When the 1929[1] tests were started there were available 12 aluminum-base alloys made by five producers and 10 zinc-base alloys made by two to six producers. Test specimens of these alloys were exposed at six outdoor locations: State College, Pa.; New York, N. Y.; Rochester, N. Y.;[3] Altoona, Pa.; Sandy Hook, N. J.; and Key West, Fla. and four indoor locations: Coco Solo Islands, Canal Zone; New Kensington, Pa.; Hanover, N. Mex.;[4] and Cambridge, Mass.

The original plans for the lengths of the test periods were changed as the exposures progressed. The aluminum-base alloys were recalled from the exposure locations and tested for their mechanical properties after 1, 5, and 15 yr. and the zinc-base alloys after 1,[5] 5, and 10 yr. except for alloy No. XXI which was only measured to determine its dimensional change after the 10-yr. period and then reexposed for an additional 5 yr. before testing its mechanical properties. Data on the mechanical properties of the aluminum-base and zinc-base alloy specimens after 15-yr. atmospheric exposures are shown in Tables I and II.

Magnesium and Zinc-Base Alloys:

The exposure tests started in 1939[2] were necessary to evaluate the merits of the newly introduced magnesium-base alloys and to serve the same purpose for the currently used zinc-base alloys since all of the zinc alloys from the 1929 tests have long since become obsolete. Test bars of four magnesium-base alloys, made by three producers, and three zinc-base alloys made by four to six producers, were prepared and exposed at the same sites used during the 1929 tests except that Tucson, Ariz., was substituted for Hanover, N. Mex. Again, the committee had an unfortunate experience at the Rochester location, only this time it was worse in that all of the specimens disappeared before any of the tests were completed.

The exposure periods have been planned for 5, 10, and an indefinite number of years. The mechanical property data for the magnesium-base and zinc-base alloy specimens after exposure for 5 yr. are given in Tables III and IV.

For more complete information on these two series of exposure programs, reference should be made to all of the committee's reports from 1928 to the present time. Since Committee B-6 was not organized until June, 1930, its earlier work is reported as that of Subcommittee XV on Die-Cast Metals and Alloys of Committee B-2 on Non-Ferrous Metals and Alloys.

Respectfully submitted on behalf of the subcommittee,

W. M. Peirce,
Chairman.

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[1] *Proceedings,* Am. Soc. Testing Mats., Vol. 29, p. 192 (1929).

[2] *Proceedings,* Am. Soc. Testing Mats., Vol. 39, p. 280 (1939).

[3] Most of the specimens at this location were missing when it was time to recall them for mechanical testing.

[4] Alloy No. XXI was the only zinc-base alloy exposed longer than 10 yr. The specimens of this alloy, which were exposed at Hanover, N. Mex. for 10 yr., had an additional exposure of 5 yr. at Tucson, Ariz.

[5] No specimens for the 1-yr. test were exposed at State College, Pa.

TABLE I.—MECHANICAL PROPERTY DATA ON ALUMINUM-BASE DIE-CASTING ALLOY SPECIMENS AFTER 15-YEAR ATMOSPHERIC EXPOSURE.

Alloy	Producer	Exposure[a]		Tensile Strength, psi.	Charpy Impact Strength, ft-lb. (¼ by ¼-in. Bars)	Elongation in 2 in., per cent	Brinell Hardness
		Type	Location				
No. I	C	Outdoors	Key West, Fla.	29 300	4	3	54
			Sandy Hook, N. J.	31 800	4	4	66 (R)
			New York, N. Y.	29 500	4	4	70 (R)
			Altoona, Pa.	29 600	4	4	64 (R)
			State College, Pa.	[b]	3	[b]	52
		Indoors	Coco Solo Islands, C. Z.	34 100	4	4	[c]
			New Kensington, Pa.	31 200	4	4	66 (R)
			Cambridge, Mass.	31 100	4	3	[c]
			Hanover, N. Mex.	31 700	5	5	[c]
No. I	D	Outdoors	Key West, Fla.	25 800	5	3	57
			Sandy Hook, N. J.	28 500	5	4	66 (R)
			New York, N. Y.	28 300	5	4	71 (R)
			Altoona, Pa.	24 500	5	3	69 (R)
			State College, Pa.	28 800	5	4	54
		Indoors	Coco Solo Islands, C. Z.	32 500	6	3	[c]
			New Kensington, Pa.	29 600	6	4	72 (R)
			Cambridge, Mass.	27 600	7	3	[c]
			Hanover, N. Mex.	27 700	6	4	[c]
No. I	G	Outdoors	Key West, Fla.	32 400	3	2	63
			Sandy Hook, N. J.	31 100	4	2	67 (R)
			New York, N. Y.	30 700	3	3	74 (R)
			Altoona, Pa.	29 700	4	2	67 (R)
			State College, Pa.	32 000	4	3	57
		Indoors	Coco Solo Islands, C. Z.	39 600	6	4	[c]
			New Kensington, Pa.	32 100	3	3	75 (R)
			Cambridge, Mass.	33 000	4	3	[c]
			Hanover, N. Mex.	31 300	5	4	[c]
No. I	S	Outdoors	Key West, Fla.	34 800	5	3	67
			Sandy Hook, N. J.	34 000	6	3	79 (R)
			New York, N. Y.	33 000	5	4	76 (R)
			Altoona, Pa.	33 800	5	4	75 (R)
			State College, Pa.	34 200	6	4	62
		Indoors	Coco Solo Islands, C. Z.	[b]	6	[b]	[c]
			New Kensington, Pa.	37 200	6	5	80 (R)
			Cambridge, Mass.	37 800	7	4	[c]
			Hanover, N. Mex.	35 600	7	5	[c]
No. I	W	Outdoors	Key West, Fla.	32 800	5	4	67
			Sandy Hook, N. J.	33 300	5	3	73 (R)
			New York, N. Y.	32 300	4	4	71 (R)
			Altoona, Pa.	32 200	5	4	75 (R)
			State College, Pa.	35 400	5	4	60
		Indoors	Coco Solo Islands, C. Z.	36 100	5	3	[c]
			New Kensington, Pa.	32 900	6	4	77 (R)
			Cambridge, Mass.	35 300	5	3	[c]
			Hanover, N. Mex.	33 500	6	4	[c]
No. II	C	Indoors	Coco Solo Islands, C. Z.	40 700	3	2	[c]
			New Kensington, Pa.	38 800	3	3	85 (R)
			Cambridge, Mass.	38 900	3	2	[c]
			Hanover, N. Mex.	37 300	3	2	[c]
No. II	D	Indoors	Coco Solo Islands, C. Z.	37 100	2	1	[c]
			New Kensington, Pa.	35 300	2	1	93 (R)
			Cambridge, Mass.	35 000	2	1	[c]
			Hanover, N. Mex.	36 000	2	1	[c]
No. II	G	Indoors	Coco Solo Islands, C. Z.	39 600	3	1	[c]
			New Kensington, Pa.	38 100	3	1	91 (R)
			Cambridge, Mass.	39 500	3	2	[c]
			Hanover, N. Mex.	37 700	4	2	[c]

[a] All specimens exposed at Rochester, N. Y., were missing. All Hanover, N. Mex., specimens were exposed for their last five years at Tucson, Ariz.

[b] Specimens were missing at the exposure location.

[c] No hardness tests were made.

(R)—Rockwell hardness, E Scale (⅛-in. ball, 100-kg. load).

TABLE I.—MECHANICAL PROPERTY DATA ON ALUMINUM-BASE DIE-CASTING ALLOY SPECIMENS AFTER 15-YEAR ATMOSPHERIC EXPOSURE.—*Continued.*

Alloy	Producer	Exposure[a]		Tensile Strength, psi.	Charpy Impact Strength, ft.-lb. (¼ by ¼-in. Bars)	Elongation in 2 in., per cent	Brinell Hardness
		Type	Location				
No. II	S	Indoors	Coco Solo Islands, C. Z.	41 700	2	1	[c]
			New Kensington, Pa.	40 500	2	1	95 (R)
			Cambridge, Mass.	40 300	2	1	[c]
			Hanover, N. Mex.	39 900	3	2	[c]
No. II	W	Indoors	Coco Solo Islands, C. Z.	45 100	2	1	[c]
			New Kensington, Pa.	38 800	2	1	95 (R)
			Cambridge, Mass.	39 000	2	1	[c]
			Hanover, N. Mex.	39 800	2	1	[c]
No. III	C	Indoors	Coco Solo Islands, C. Z.	43 600	1	1	[c]
			New Kensington, Pa.	41 400	1	1	94 (R)
			Cambridge, Mass.	43 100	1	1	[c]
			Hanover, N. Mex.	40 400	2	2	[c]
No. III	D	Indoors	Coco Solo Islands, C. Z.	42 100	1	1	[c]
			New Kensington, Pa.	40 400	1	1	102 (R)
			Cambridge, Mass.	40 700	1	0	[c]
			Hanover, N. Mex.	38 500	1	1	[c]
No. III	G	Indoors	Coco Solo Islands, C. Z.	40 200	1	1	[c]
			New Kensington, Pa.	40 900	1	1	98 (R)
			Cambridge, Mass.	39 500	1	1	[c]
			Hanover, N. Mex.	38 500	2	1	[c]
No. III	S	Indoors	Coco Solo Islands, C. Z.	44 200	1	1	[c]
			New Kensington, Pa.	41 600	2	1	100 (R)
			Cambridge, Mass.	43 700	1	1	[c]
			Hanover, N. Mex.	41 900	2	1	[c]
No. III	W	Indoors	Coco Solo Islands, C. Z.	43 700	1	1	[c]
			New Kensington, Pa.	43 000	1	0	103 (R)
			Cambridge, Mass.	43 800	1	1	[c]
			Hanover, N. Mex.	39 200	1	1	[c]
No. IV	C	Outdoors	Key West, Fla.	30 600	4	4	53
			Sandy Hook, N. J.	30 800	4	3	63 (R)
			New York, N. Y.	30 400	4	4	67 (R)
			Altoona, Pa.	29 400	4	4	60 (R)
			State College, Pa.	31 900	4	3	52
		Indoors	Coco Solo Islands, C. Z.	33 000	3	4	[c]
			New Kensington, Pa.	31 500	4	4	59 (R)
			Cambridge, Mass.	32 500	5	4	[c]
			Hanover, N. Mex.	31 800	5	5	[c]
No. IV	D	Outdoors	Key West, Fla.	25 400	4	3	54
			Sandy Hook, N. J.	26 200	3	3	61 (R)
			New York, N. Y.	26 100	3	2	67 (R)
			Altoona, Pa.	24 300	3	2	61 (R)
			State College, Pa.	27 300	3	2	49
		Indoors	Coco Solo Islands, C. Z.	27 400	4	2	[c]
			New Kensington, Pa.	25 500	4	2	66 (R)
			Cambridge, Mass.	26 700	4	3	[c]
			Hanover, N. Mex.	26 100	4	3	[c]
No. IV	G	Outdoors	Key West, Fla.	30 800	5	4	56
			Sandy Hook, N. J.	30 000	5	3	65 (R)
			New York, N. Y.	29 100	4	3	69 (R)
			Altoona, Pa.	29 200	4	4	63 (R)
			State College, Pa.	30 400	4	3	52
		Indoors	Coco Solo Islands, C. Z.	31 100	5	3	[c]
			New Kensington, Pa.	30 300	5	4	68 (R)
			Cambridge, Mass.	31 100	5	3	[c]
			Hanover, N. Mex.	30 000	5	4	[c]
No. IV	S	Outdoors	Key West, Fla.	29 300	5	4	59
			Sandy Hook, N. J.	29 500	5	4	69 (R)
			New York, N. Y.	28 000	4	3	82 (R)
			Altoona, Pa.	29 800	4	4	67 (R)
			State College, Pa.	32 100	5	4	50

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[a] All specimens exposed at Rochester, N. Y., were missing. All Hanover, N. Mex., specimens were exposed for the last five years at Tucson, Ariz.

[c] No hardness tests were made.

(R)—Rockwell hardness, E Scale (⅛-in. ball, 100-kg. load).

TABLE I.—MECHANICAL PROPERTY DATA ON ALUMINUM-BASE DIE-CASTING ALLOY SPECIMENS AFTER 15-YEAR ATMOSPHERIC EXPOSURE.—*Continued.*

Alloy	Producer	Exposure[a]		Tensile Strength, psi.	Charpy Impact Strength, ft-lb. (¼ by ¼-in. Bars)	Elongation in 2 in., per cent	Brinell Hardness
		Type	Location				
		Indoors	Coco Solo Islands, C. Z.	32 500	6	3	[c]
			New Kensington, Pa.	31 900	6	4	64 (R)
			Cambridge, Mass.	31 000	6	3	[c]
			Hanover, N. Mex.	29 600	6	4	[c]
No. IV	W	Outdoors	Key West, Fla.	32 300	2	2	76
			Sandy Hook, N. J.	30 300	1	1	87 (R)
			New York, N. Y.	31 300	2	2	75 (R)
			Altoona, Pa.	32 200	2	1	82 (R)
			State College, Pa.	34 400	2	1	70
		Indoors	Coco Solo Islands, C. Z.	32 900	2	1	[c]
			New Kensington, Pa.	32 700	2	1	83 (R)
			Cambridge, Mass.	31 200	2	1	[c]
			Hanover, N. Mex.	31 400	2	2	[c]
No. V	C	Outdoors	Key West, Fla.	34 200	2	1	78
			Sandy Hook, N. J.	32 300	1	1	80 (R)
			New York, N. Y.	29 500	1	1	86 (R)
			Altoona, Pa.	31 700	1	2	81 (R)
			State College, Pa.	33 400	1	2	76
		Indoors	Coco Solo Islands, C. Z.	34 400	2	1	[c]
			New Kensington, Pa.	34 600	1	2	79 (R)
			Cambridge, Mass.	33 900	2	1	[c]
			Hanover, N. Mex.	32 800	2	2	[c]
No. V	D	Outdoors	Key West, Fla.	26 400	1	1	76
			Sandy Hook, N. J.	25 600	1	1	76 (R)
			New York, N. Y.	26 300	1	1	85 (R)
			Altoona, Pa.	24 600	1	2	80 (R)
			State College, Pa.	26 300	1	1	74
		Indoors	Coco Solo Islands, C. Z.	27 600	2	1	[c]
			New Kensington, Pa.	27 700	1	1	83 (R)
			Cambridge, Mass.	25 400	2	1	[c]
			Hanover, N. Mex.	26 200	2	1	[c]
No. V	G	Outdoors	Key West, Fla.	27 000	2	1	69
			Sandy Hook, N. J.	26 100	1	1	79 (R)
			New York, N. Y.	24 900	1	1	81 (R)
			Altoona, Pa.	24 600	1	2	80 (R)
			State College, Pa.	27 700	1	1	60
		Indoors	Coco Solo Islands, C. Z.	29 700	2	1	[c]
			New Kensington, Pa.	30 400	1	2	81 (R)
			Cambridge, Mass.	30 300	2	1	[c]
			Hanover, N. Mex.	27 600	1	2	[c]
No. V	S	Outdoors	Key West, Fla.	35 200	3	2	79
			Sandy Hook, N. J.	33 900	2	1	76 (R)
			New York, N. Y.	35 600	2	2	92 (R)
			Altoona, Pa.	33 900	2	2	80 (R)
			State College, Pa.	37 300	2	2	64
		Indoors	Coco Solo Islands, C. Z.	36 800	[b]	2	[b]
			New Kensington, Pa.	36 400	3	2	82 (R)
			Cambridge, Mass.	36 800	4	2	[c]
			Hanover, N. Mex.	36 600	3	2	[c]
No. V	W	Outdoors	Key West, Fla.	30 300	1	1	80
			Sandy Hook, N. J.	28 600	1	0	83 (R)
			New York, N. Y.	29 500	1	1	82 (R)
			Altoona, Pa.	27 800	1	1	87 (R)
			State College, Pa.	31 500	1	1	83
		Indoors	Coco Solo Islands, C. Z.	30 000	1	1	[c]
			New Kensington, Pa.	28 200	1	1	92 (R)
			Cambridge, Mass.	30 700	1	1	[c]
			Hanover, N. Mex.	26 100	1	1	[c]

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[a] All specimens exposed at Rochester, N. Y., were missing. All Hanover, N. Mex., specimens were exposed for their last five years at Tucson, Ariz.

[b] Specimens were missing at the exposure location.

[c] No hardness tests were made.

(R)—Rockwell hardness, E Scale (⅛-in. ball, 100-kg. load).

TABLE I.—MECHANICAL PROPERTY DATA ON ALUMINUM-BASE DIE-CASTING ALLOY SPECIMENS AFTER 15-YEAR ATMOSPHERIC EXPOSURE.—*Continued.*

Alloy	Producer	Exposure[a]		Tensile Strength, psi.	Charpy Impact Strength, ft-lb. (¼ by ¼-in. Bars)	Elongation in 2 in., per cent	Brinell Hardness
		Type	Location				
No. VI	C	Indoors	Coco Sclo Islands, C. Z.	31 900	3	3	[c]
			New Kensington, Pa.	34 000	3	4	74 (R)
			Cambridge, Mass.	31 800	4	3	[c]
			Hanover, N. Mex.	32 600	4	4	[c]
No. VI	D	Indoors	Coco Solo Islands, C. Z.	28 300	4	2	[c]
			New Kensington, Pa.	30 200	4	3	69 (R)
			Cambridge, Mass.	29 800	4	3	[c]
			Hanover, N. Mex.	26 800	5	3	[c]
No. VI	G	Indoors	Coco Solo Islands, C. Z.	33 200	5	3	[c]
			New Kensington, Pa.	33 500	4	4	73 (R)
			Cambridge, Mass.	32 900	5	4	[c]
			Hanover, N. Mex.	32 900	4	4	[c]
No VI	S	Indoors	Coco Solo Islands, C. Z.	33 900	7	3	[c]
			New Kensington, Pa.	33 200	5	4	74 (R)
			Cambridge, Mass.	34 700	6	4	[c]
			Hanover, N. Mex.	33 400	6	4	[c]
No. VI	W	Indoors	Coco Solo Islands, C. Z.	32 800	2	1	[c]
			New Kensington, Pa.	34 500	2	2	85 (R)
			Cambridge, Mass.	34 000	2	2	[c]
			Hanover, N. Mex.	34 400	2	2	[c]
No. VII	C	Outdoors	Key West, Fla.	34 700	3	2	72
			Sandy Hook, N. J.	36 800	2	2	76 (R)
			New York, N. Y.	36 700	2	3	78 (R)
			Altoona, Pa.	34 800	2	3	79 (R)
			State College, Pa.	39 100	2	3	68
		Indoors	Coco Solo Islands, C. Z.	38 500	3	3	[c]
			New Kensington, Pa.	37 200	2	3	79 (R)
			Cambridge, Mass.	38 800	3	3	[c]
			Hanover, N. Mex.	37 800	3	3	[c]
No. VII	D	Outdoors	Key West, Fla.	29 800	1	1	72
			Sandy Hook, N. J.	28 200	1	0	84 (R)
			New York, N. Y.	29 800	1	1	78 (R)
			Altoona, Pa.	29 000	1	1	88 (R)
			State College, Pa.	31 000	1	1	80
		Indoors	Coco Solo Islands, C. Z.	28 400	2	0	[c]
			New Kensington, Pa.	30 700	2	1	88 (R)
			Cambridge, Mass.	29 100	2	1	[c]
			Hanover, N. Mex.	28 800	2	1	[c]
No. VII	G	Outdoors	Key West, Fla.	30 200	2	2	76
			Sandy Hook, N. J.	31 100	2	1	81 (R)
			New York, N. Y.	30 900	2	1	81 (R)
			Altoona, Pa.	30 500	1	1	85 (R)
			State College, Pa.	32 700	1	2	69
		Indoors	Coco Solo Islands, C. Z.	32 800	2	1	[c]
			New Kensington, Pa.	32 200	2	2	84 (R)
			Cambridge, Mass.	32 700	3	1	[c]
			Hanover, N. Mex.	33 800	3	2	[c]
No. VII	S	Outdoors	Key West, Fla.	35 600	1	1	84
			Sandy Hook, N. J.	36 400	1	1	88 (R)
			New York, N. Y.	35 700	1	1	90 (R)
			Altoona, Pa.	34 700	1	2	91 (R)
			State College, Pa.	36 900	2	1	87
		Indoors	Coco Solo Islands, C. Z.	36 600	[b]	1	[b]
			New Kensington, Pa.	37 200	2	2	91 (R)
			Cambridge, Mass.	37 900	2	1	[c]
			Hanover, N. Mex.	36 200	2	1	[c]

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[a] All specimens exposed at Rochester, N. Y., were missing. All Hanover, N. Mex., specimens were exposed for thei last five years at Tucson, Ariz.

[b] Specimens were missing at the exposure location.

[c] No hardness tests were made.

(R)—Rockwell hardness, E Scale (⅛-in. ball, 100-kg. load).

TABLE I.—MECHANICAL PROPERTY DATA ON ALUMINUM-BASE DIE-CASTING ALLOY SPECIMENS AFTER 15-YEAR ATMOSPHERIC EXPOSURE.—*Continued.*

Alloy	Producer	Exposure[a]		Tensile Strength, psi.	Charpy Impact Strength, ft-lb. (¼ by ¼-Bars)	Elongation in 2 in., per cent	Brinell Hardness
		Type	Location				
No. VII	W	Outdoors	Key West, Fla.	32 800	1	1	91
			Sandy Hook, N. J.	30 400	1	0	91 (R)
			New York, N. Y.	33 400	1	1	91 (R)
			Altoona, Pa.	33 600	1	1	92 (R)
			State College, Pa.	32 300	1	1	92
		Indoors	Coco Solo Islands, C. Z.	35 300	1	1	[c]
			New Kensington, Pa.	35 500	1	1	94 (R)
			Cambridge, Mass.	34 300	1	1	[c]
			Hanover, N. Mex.	33 800	1	1	[c]
No. VIII	C	Outdoors	Key West, Fla.	32 900	4	6	49
			Sandy Hook, N. J.	31 300	3	4	58 (R)
			New York, N. Y.	31 800	4	7	61 (R)
			Altoona, Pa.	32 000	3	6	61 (R)
			State College, Pa.	33 100	4	6	46
		Indoors	Coco Solo Islands, C. Z.	34 600	4	6	[c]
			New Kensington, Pa.	33 500	4	6	58 (R)
			Cambridge, Mass.	32 100	5	5	[c]
			Hanover, N. Mex.	33 500	5	7	[c]
No. VIII	D	Outdoors	Key West, Fla.	28 500	3	3	58
			Sandy Hook, N. J.	25 900	3	2	67 (R)
			New York, N. Y.	28 300	2	3	70 (R)
			Altoona, Pa.	23 500	3	2	67 (R)
			State College, Pa.	26 900	2	2	56
		Indoors	Coco Solo Islands, C. Z.	28 400	3	2	
			New Kensington, Pa.	27 800	3	2	72 (R)
			Cambridge, Mass.	28 500	3	2	[c]
			Hanover, N. Mex.	27 100	4	3	[c]
No. VIII	G	Outdoors	Key West, Fla.	29 600	4	4	58
			Sandy Hook, N. J.	28 500	4	3	64 (R)
			New York, N. Y.	28 600	4	4	63 (R)
			Altoona, Pa.	28 100	3	4	67 (R)
			State College, Pa.	29 400	3	3	51
		Indoors	Coco Solo Islands, C. Z.	27 500	4	3	[c]
			New Kensington, Pa.	28 800	4	3	64 (R)
			Cambridge, Mass.	28 300	5	3	[c]
			Hanover, N. Mex.	28 200	5	4	[c]
No. VIII	S	Outdoors	Key West, Fla.	35 100	5	5	61
			Sandy Hook, N. J.	34 500	4	4	71 (R)
			New York, N. Y.	34 200	4	5	70 (R)
			Altoona, Pa.	33 700	4	5	71 (R)
			State College, Pa.	35 300	5	5	56
		Indoors	Coco Solo Islands, C. Z.	36 800	4	5	[c]
			New Kensington, Pa.	35 500	4	5	72 (R)
			Cambridge, Mass.	37 100	5	6	[c]
			Hanover, N. Mex.	34 900	5	5	[c]
No. VIII	W	Outdoors	Key West, Fla.	30 600	6	5	49
			Sandy Hook, N. J.	31 500	5	4	62 (R)
			New York, N. Y.	28 100	5	4	60 (R)
			Altoona, Pa.	26 100	5	4	61 (R)
			State College, Pa.	27 900	4	4	49
		Indoors	Coco Solo Islands, C. Z.	32 300	6	6	
			New Kensington, Pa.	31 800	6	5	64 (R)
			Cambridge, Mass.	31 700	6	5	[c]
			Hanover, N. Mex.	31 000	7	6	[c]
No. IX	C	Outdoors	Key West, Fla.	38 100	1	2	90
			Sandy Hook, N. J.	36 400	1	1	92 (R)
			New York, N. Y.	37 100	1	1	94 (R)
			Altoona, Pa.	34 700	1	1	94 (R)
			State College, Pa.	38 700	1	1	89

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[a] All specimens exposed at Rochester, N. Y., were missing. All Hanover, N. Mex., specimens were exposed for their last five years at Tucson, Ariz.

[c] No hardness tests were made.

(R)—Rockwell hardness, E Scale (⅛-in. ball, 100-kg. load).

TABLE I.—MECHANICAL PROPERTY DATA ON ALUMINUM-BASE DIE-CASTING ALLOY SPECIMENS AFTER 15-YEAR ATMOSPHERIC EXPOSURE.—*Continued.*

Alloy	Producer	Exposure[a]		Tensile Strength, psi.	Charpy Impact Strength, ft-lb. (¼ by ¼-in. Bars)	Elongation in 2 in., per cent	Brinell Hardness
		Type	Location				
		Indoors	Coco Solo Islands, C. Z.	39 900	1	1	[c]
			New Kensington, Pa.	38 500	1	1	92 (R)
			Cambridge, Mass.	35 000	2	1	[c]
			Hanover, N. Mex.	37 500	1	1	[c]
No. IX	D	Outdoors	Key West, Fla.	27 600	1	1	76
			Sandy Hook, N. J.	28 000	1	1	81 (R)
			New York, N. Y.	27 500	1	1	82 (R)
			Altoona, Pa.	25 500	1	1	82 (R)
			State College, Pa.	28 800	1	1	69
		Indoors	Coco Solo Islands, C. Z.	28 900	2	1	[c]
			New Kensington, Pa.	28 800	1	1	81 (R)
			Cambridge, Mass.	30 400	2	1	[c]
			Hanover, N. Mex.	27 200	2	1	[c]
No. IX	G	Outdoors	Key West, Fla.	33 700	1	2	77
			Sandy Hook, N. J.	32 300	2	1	85 (R)
			New York, N. Y.	32 600	1	2	86 (R)
			Altoona, Pa.	32 100	1	2	85 (R)
			State College, Pa.	33 400	1	2	71
		Indoors	Coco Solo Islands, C. Z.	35 000	2	1	[c]
			New Kensington, Pa.	35 300	2	2	88 (R)
			Cambridge, Mass.	34 200	2	2	[c]
			Hanover, N. Mex.	35 000	2	2	[c]
No. IX	S	Outdoors	Key West, Fla.	38 000	2	2	74
			Sandy Hook, N. J.	37 500	2	1	86 (R)
			New York, N. Y.	37 100	2	2	87 (R)
			Altoona, Pa.	36 200	1	2	87 (R)
			State College, Pa.	39 100	2	2	74
		Indoors	Coco Solo Islands, C. Z.	41 300	2	2	[c]
			New Kensington, Pa.	38 800	2	2	89 (R)
			Cambridge, Mass.	40 300	2	2	[c]
			Hanover, N. Mex.	38 300	2	2	[c]
No. IX	W	Outdoors	Key West, Fla.	38 800	1	2	92
			Sandy Hook, N. J.	39 000	1	1	93 (R)
			New York, N. Y.	39 300	1	1	94 (R)
			Altoona, Pa.	36 900	1	1	93 (R)
			State College, Pa.	39 500	1	2	93
		Indoors	Coco Solo Islands, C. Z.	40 800	2	1	
			New Kensington, Pa.	40 100	2	2	95 (R)
			Cambridge, Mass.	40 600	2	2	[c]
			Hanover, N. Mex.	39 800	2	2	[c]
No. X	C	Outdoors	Key West, Fla.	34 000	3	3	63
			Sandy Hook, N. J.	32 400	2	2	73 (R)
			New York, N. Y.	34 000	2	3	71 (R)
			Altoona, Pa.	33 500	3	3	75 (R)
			State College, Pa.	34 600	2	3	55
		Indoors	Coco Solo Islands, C. Z.	35 600	3	2	[c]
			New Kensington, Pa.	32 900	3	2	71 (R)
			Cambridge, Mass.	33 800	3	2	[c]
			Hanover, N. Mex.	33 400	3	3	[c]
No. X	D	Outdoors	Key West, Fla.	23 200	2	2	53
			Sandy Hook, N. J.	24 400	2	2	60 (R)
			New York, N. Y.	22 800	2	1	63 (R)
			Altoona, Pa.	22 500	2	2	65 (R)
			State College, Pa.	24 900	1	2	51
		Indoors	Coco Solo Islands, C. Z.	23 800	2	1	
			New Kensington, Pa.	23 600	2	2	62 (R)
			Cambridge, Mass.	24 800	2	2	[c]
			Hanover, N. Mex.	23 600	2	2	[c]

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[a] All specimens exposed at Rochester, N. Y., were missing. All Hanover, N. Mex., specimens were exposed for the last five years at Tucson, Ariz.

[c] No hardness tests were made.

(R)—Rockwell hardness, E Scale (⅛-in. ball, 100 kg. load).

TABLE I.—MECHANICAL PROPERTY DATA ON ALUMINUM-BASE DIE-CASTING ALLOY SPECIMENS AFTER 15-YEAR ATMOSPHERIC EXPOSURE—*Continued.*

Alloy	Producer	Exposure[a]		Tensile Strength, psi.	Charpy Impact Strength, ft-lb. (¼ by ¼-in. Bars)	Elongation in 2 in., per cent	Brinell Hardness
		Type	Location				
No. X	G	Outdoors	Key West, Fla.	28 100	2	2	55
			Sandy Hook, N. J.	28 400	2	2	70 (R)
			New York, N. Y.	27 500	2	2	72 (R)
			Altoona, Pa.	26 900	2	2	71 (R)
			State College, Pa.	27 700	2	2	54
		Indoors	Coco Solo Islands, C. Z.	27 900	2	2	[c]
			New Kensington, Pa.	28 700	2	3	70 (R)
			Cambridge, Mass.	28 100	2	2	[c]
			Hanover, N. Mex.	28 300	3	2	[c]
No. X	S	Outdoors	Key West, Fla	31 600	3	3	63
			Sandy Hook, N. J.	31 900	3	2	71 (R)
			New York, N. Y.	31 500	3	3	79 (R)
			Altoona, Pa.	30 800	2	3	71 (R)
			State College, Pa.	33 400	3	3	62
		Indoors	Coco Solo Islands, C. Z.	33 400	3	3	[c]
			New Kensington, Pa.	32 900	3	3	74 (R)
			Cambridge, Mass.	32 700	3	3	[c]
			Hanover, N. Mex.	32 300	3	3	[c]
No. X	W	Outdoors	Key West, Fla.	29 500	3	2	63
			Sandy Hook, N. J.	30 200	2	2	70 (R)
			New York, N. Y.	26 600	2	2	70 (R)
			Altoona, Pa.	27 700	2	2	71 (R)
			State College, Pa.	30 900	2	2	61
		Indoors	Coco Solo Islands, C. Z.	30 300	3	2	[c]
			New Kensington, Pa.	30 400	3	2	71 (R)
			Cambridge, Mass.	30 500	3	2	[c]
			Hanover, N. Mex.	29 700	3	3	[c]
No. XI	C	Outdoors	Key West, Fla.	36 100	2	3	71
			Sandy Hook, N. J.	35 900	2	2	77 (R)
			New York, N. Y.	34 500	2	3	84 (R)
			Altoona, Pa.	35 200	2	3	76 (R)
			State College, Pa.	38 600	2	3	69
		Indoors	Coco Solo Islands, C. Z.	39 100	3	3	[c]
			New Kensington, Pa.	38 300	3	3	77 (R)
			Cambridge, Mass.	38 700	3	3	[c]
			Hanover, N. Mex.	37 100	3	3	[c]
No. XI	D	Outdoors	Key West, Fla.	29 000	3	3	65
			Sandy Hook, N. J.	26 600	3	1	73 (R)
			New York, N. Y.	28 100	3	2	79 (R)
			Altoona, Pa.	29 100	3	2	72 (R)
			State College, Pa.	30 100	3	2	64
		Indoors	Coco Solo Islands, C. Z.	30 400	4	2	[c]
			New Kensington, Pa.	27 500	3	2	76 (R)
			Cambridge, Mass.	27 600	4	2	[c]
			Hanover, N. Mex.	27 500	4	2	[c]
No. XI	G	Outdoors	Key West, Fla.	32 600	2	3	70
			Sandy Hook, N. J.	32 300	2	2	78 (R)
			New York, N. Y.	33 000	2	2	84 (R)
			Altoona, Pa.	31 200	2	2	78 (R)
			State College, Pa.	33 900	2	2	68
		Indoors	Coco Solo Islands, C. Z.	33 000	3	1	[c]
			New Kensington, Pa.	33 200	2	2	81 (R)
			Cambridge, Mass.	34 100	3	2	[c]
			Hanover, N. Mex	33 000	3	2	[c]
No. XI	S	Outdoors	Key West, Fla.	34 300	2	2	84
			Sandy Hook, N. J.	33 500	1	1	84 (R)
			New York, N. Y.	33 500	2	2	90 (R)
			Altoona, Pa.	33 200	1	2	88 (R)
			State College, Pa.	36 200	2	2	81

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[a] All specimens exposed at Rochester, N. Y., were missing. All Hanover, N. Mex., specimens were exposed for their last five years at Tucson, Ariz.

[c] No hardness tests were made.

(R)—Rockwell hardness, E Scale (⅛-in. ball, 100-kg. load).

TABLE I.—MECHANICAL PROPERTY DATA ON ALUMINUM-BASE DIE-CASTING ALLOY SPECIMENS AFTER 15-YEAR ATMOSPHERIC EXPOSURE.—*Concluded.*

Alloy	Producer	Exposure[a]		Tensile Strength, psi.	Charpy Impact Strength, ft-lb. (¼ by ¼-in. Bars)	Elongation in 2 in., per cent	Brinell Hardness
		Type	Location				
		Indoors	Coco Solo Islands, C. Z.	35 400	2	1	[c]
			New Kensington, Pa.	35 900	2	2	89 (R)
			Cambridge, Mass.	36 000	2	2	[c]
			Hanover, N. Mex.	35 000	2	2	[c]
No. XI	W	Outdoors	Key West, Fla.	26 900	1	1	79
			Sandy Hook, N. J.	28 300	1	1	87 (R)
			New York, N. Y.	30 300	1	1	86 (R)
			Altoona, Pa.	28 700	1	1	86 (R)
			State College, Pa.	31 300	1	1	77
		Indoors	Coco Solo Islands, C. Z.	30 100	1	1	[c]
			New Kensington, Pa.	29 200	1	1	88 (R)
			Cambridge, Mass.	31 200	2	1	[c]
			Hanover, N. Mex.	29 600	1	1	[c]
No. XII	C	Outdoors	Key West, Fla.	36 400	2	2	80
			Sandy Hook, N. J.	35 800	2	2	84 (R)
			New York, N. Y.	36 600	2	2	85 (R)
			Altoona, Pa.	34 800	2	2	87 (R)
			State College, Pa.	38 000	2	1	82
		Indoors	Coco Solo Islands, C. Z.	39 800	2	2	[c]
			New Kensington, Pa.	39 700	3	2	85 (R)
			Cambridge, Mass.	39 400	3	2	[c]
			Hanover, N. Mex.	37 700	2	2	[c]
No. XII	D	Outdoors	Key West, Fla.	29 400	1	1	84
			Sandy Hook, N. J.	28 000	1	1	83 (R)
			New York, N. Y.	32 500	1	1	91 (R)
			Altoona, Pa.	30 900	1	1	90 (R)
			State College, Pa.	33 600	1	1	88
		Indoors	Coco Solo Islands, C. Z.	34 300	2	0	[c]
			New Kensington, Pa.	35 800	2	1	92 (R)
			Cambridge, Mass.	34 400	2	1	[c]
			Hanover, N. Mex.	33 900	2	1	[c]
No. XII	G	Outdoors	Key West, Fla.	25 400	2	1	80
			Sandy Hook, N. J.	32 700	2	1	76 (R)
			New York, N. Y.	31 400	2	2	86 (R)
			Altoona, Pa.	29 300	2	1	87 (R)
			State College, Pa.	31 900	2	1	70
		Indoors	Coco Solo Islands, C. Z.	31 400	2	1	[c]
			New Kensington, Pa.	33 000	2	2	85 (R)
			Cambridge, Mass.	35 200	2	1	[c]
			Hanover, N. Mex.	34 700	2	2	[c]
No. XII	S	Outdoors	Key West, Fla.	31 200	2	2	96
			Sandy Hook, N. J.	31 000	2	1	85 (R)
			New York, N. Y.	37 300	2	2	92 (R)
			Altoona, Pa.	37 500	2	2	92 (R)
			State College, Pa.	38 500	2	2	91
		Indoors	Coco Solo Islands, C. Z.	40 600	3	1	[c]
			New Kensington, Pa.	41 600	2	2	91 (R)
			Cambridge, Mass.	42 200	3	2	[c]
			Hanover, N. Mex.	39 700	3	2	[c]
No. XII	W	Outdoors	Key West, Fla.	35 800	1	2	91
			Sandy Hook, N. J.	30 700	1	1	90 (R)
			New York, N. Y.	35 400	1	1	91 (R)
			Altoona, Pa.	34 600	1	2	94 (R)
			State College, Pa.	38 600	1	1	96
		Indoors	Coco Solo Islands, C. Z.	40 200	2	1	[c]
			New Kensington, Pa.	40 700	2	2	97 (R)
			Cambridge, Mass.	37 600	2	1	[c]
			Hanover, N. Mex.	38 500	1	2	[c]

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[a] All specimens exposed at Rochester, N. Y., were missing. All Hanover, N. Mex., specimens were exposed for their last five years at Tucson, Ariz.

[c] No hardness tests were made.

(R)—Rockwell hardness, E Scale (⅛-in. ball, 100-kg. load).

TABLE II.—MECHANICAL PROPERTY DATA ON ZINC-BASE DIE-CASTING ALLOY SPECIMENS AFTER 15-YEAR ATMOSPHERIC EXPOSURE.

Alloy	Producer	Exposure[a]		Tensile Strength, psi.	Charpy Impact Strength, ft-lb. (¼ by ¼-in. Bars)	Elongation in 2 in., per cent	Brinell Hardness	Expansion, in. per 6 in.
		Type	Location					
No. XXI	D	Outdoors	Key West, Fla.	43 900	1	1	94	0.0151
			Sandy Hook, N. J.	47 900	1	0	91 (R)	0.0103
			New York, N. Y.	44 300	2	1	94 (R)	0.0060
			Altoona, Pa.	41 200	2	1	97 (R)	0.0043
			State College, Pa.	49 000	1	1	99 (R)	0.0083
		Indoors	Coco Solo Islands, C. Z.	44 200	[b]	1	[b]	[b]
			New Kensington, Pa.	44 900	3	3	95 (R)	0.0116
			Cambridge, Mass.	47 500	1	2	77	0.0108
			Hanover, N. Mex.	49 000	1	1	94 (R)	0.0116
No. XXI	R	Outdoors	Key West, Fla.	42 500	1	1	93	0.0133
			Sandy Hook, N. J.	50 800	1	0	85 (R)	0.0065
			New York, N. Y.	45 700	2	1	97 (R)	0.0022
			Altoona, Pa.	43 900	2	2	94 (R)	−0.0010
			State College, Pa.	51 700	1	1	100 (R)	0.0037
		Indoors	Coco Solo Islands, C. Z.	44 300	1	1	90	0.0129
			New Kensington, Pa.	44 900	1	1	95 (R)	0.0112
			Cambridge, Mass.	48 500	1	2	76	0.0097
			Hanover, N. Mex.	46 700	1	1	84 (R)	0.0104
No. XXI	S	Outdoors	Key West, Fla.	46 300	1	1	94	0.0105
			Sandy Hook, N. J.	49 500	1	1	93 (R)	0.0041
			New York, N. Y.	44 100	2	1	94 (R)	0.0016
			Altoona, Pa.	42 000	1	2	98 (R)	0.0000
			State College, Pa.	50 800	1	2	100 (R)	0.0034
		Indoors	Coco Solo Islands, C. Z.	46 500	1	1	86	0.0084
			New Kensington, Pa.	46 200	3	3	97 (R)	0.0065
			Cambridge, Mass.	50 300	2	2	84	0.0059
			Hanover, N. Mex.	49 600	1	1	95 (R)	0.0060
No. XXI	T	Outdoors	Key West, Fla.	39 700	1	1	89	0.0150
			Sandy Hook, N. J.	46 400	1	1	78 (R)	0.0102
			New York, N. Y.	42 600	2	1	93 (R)	0.0070
			Altoona, Pa.	42 200	2	3	95 (R)	0.0059
			State College, Pa.	47 500	1	1	98 (R)	0.0091
		Indoors	Coco Solo Islands, C. Z.	42 100	1	1	73	0.0137
			New Kensington, Pa.	43 909	2	2	84 (R)	0.0113
			Cambridge, Mass.	42 900	2	1	77	0.0116
			Hanover, N. Mex.	43 800	1	0	91 (R)	0.0115
No. XXI	Z	Outdoors	Key West, Fla.	37 700	1	1	88	0.0142
			Sandy Hook, N. J.	48 600	1	0	87 (R)	0.0039
			New York, N. Y.	41 300	2	1	89 (R)	0.0027
			Altoona, Pa.	41 700	1	3	93 (R)	0.0037
			State College, Pa.	49 500	1	2	96 (R)	0.0043
		Indoors	Coco Solo Islands, C. Z.	40 700	1	1	79	0.0116
			New Kensington, Pa.	42 000	2	3	92 (R)	0.0102
			Cambridge, Mass.	43 700	1	2	76	0.0088
			Hanover, N. Mex.	45 200	1	0	92 (R)	0.0097

[a] All specimens exposed at Rochester, N. Y., were missing. All Hanover, N. Mex., specimens were exposed for their last five years at Tucson, Ariz.

[b] Specimens were missing at the exposure location.

(R)—Rockwell hardness, E Scale (⅛-in. ball, 100-kg. load).

TABLE III.—MECHANICAL PROPERTIES AND EXPANSION DATA ON MAGNESIUM-BASE DIE-CASTING ALLOY SPECIMENS AFTER 5-YEAR ATMOSPHERIC EXPOSURE.

Alloy	Producer	Exposure[a]		Tensile Strength, psi.	Charpy Impact Strength, ft-lb. (¼ by ¼-in. Bars)	Elongation in 2 in., per cent	Brinell Hardness	Expansion, in. per 6 in.[b]
		Type	Location					
No. 307	H	Outdoors	Key West, Fla.	28 300	2	4	67 (R)	−0.0007
			Sandy Hook, N. J.	25 500	2	3	54	−0.0012
			New York, N. Y.	24 400	2	4	49	−0.0028
			Altoona, Pa.	24 400	2	4	52 (R)	−0.0022
			State College, Pa.	27 100	2	3	63 (R)	−0.0009
		Indoors	Coco Solo Islands, C. Z.	28 700	3	4	67 (R)	−0.0001
			New Kensington, Pa.	29 000	2	4	51	−0.0004
			Cambridge, Mass.	31 300	0.5 (N)	5	64	−0.0003
			Tucson, Ariz.	30 500	0.5 (N)	5	72	−0.0005
No. 307	K	Outdoors	Key West, Fla.	32 700	4	7	68 (R)	−0.0007
			Sandy Hook, N. J.	29 200	3	5	53	−0.0015
			New York, N. Y.	28 000	3	6	49	−0.0047
			Altoona, Pa.	29 300	3	7	59 (R)	−0.0029
			State College, Pa.	29 300	3	5	63 (R)	−0.0007
		Indoors	Coco Solo Islands, C. Z.	32 800	3	7	70 (R)	0.0002
			New Kensington, Pa.	34 800	3	8	52	−0.0001
			Cambridge, Mass.	32 500	0.7 (N)	6	84	−0.0001
			Tucson, Ariz.	34 600	0.6 (N)	8	72	−0.0002
No. 307	M	Outdoors	Key West, Fla.	25 300	3	3	64 (R)	−0.0004
			Sandy Hook, N. J.	23 400	3	2	53	−0.0019
			New York, N. Y.	21 400	3	2	51	−0.0034
			Altoona, Pa.	21 500	3	2	59 (R)	−0.0033
			State College, Pa.	24 100	3	2	64 (R)	−0.0008
		Indoors	Coco Solo Islands, C. Z.	25 100	5	3	65 (R)	−0.0002
			New Kensington, Pa.	27 200	4	...	52	−0.0002
			Cambridge, Mass.	26 000	0.6 N)	3	83	−0.0003
			Tucson, Ariz.	26 200	0.6 (N)	3	72	−0.0005
No. 312	H	Outdoors	Key West, Fla.	28 000	1	1	76 (R)	−0.0008
			Sandy Hook, N. J.	26 900	1	1	60	−0.0016
			New York, N. Y.	26 700	1	2	59	−0.0030
			Altoona, Pa.	23 900	1	1	72 (R)	−0.0024
			State College, Pa.	27 600	1	1	74 (R)	−0.0010
		Indoors	Coco Solo Islands, C. Z.	33 400	1	3	77 (R)	−0.0012
			New Kensington, Pa.	29 400	1	1	62	−0.0008
			Cambridge, Mass.	28 800	0.3 (N)	1	95	−0.0008
			Tucson, Ariz.	29 200	0.3 (N)	1	84	−0.0008
No. 312	K	Outdoors	Key West, Fla.	30 900	1	2	74 (R)	0.0000
			Sandy Hook, N. J.	29 000	1	2	62	−0.0010
			New York, N. Y.	29 400	2	3	58	−0.0037
			Altoona, Pa.	28 600	1	2	71 (R)	−0.0021
			State College, Pa.	30 400	1	2	77 (R)	−0.0004
		Indoors	Coco Solo Islands, C. Z.	34 300	1	4	81 (R)	0.0001
			New Kensington, Pa.	32 100	1	3	60	−0.0001
			Cambridge, Mass.	32 600	0.3 (N)	2	95	−0.0002
			Tucson, Ariz.	32 800	0.3 (N)	3	82	0.0000
No. 312	M	Outdoors	Key West, Fla.	30 300	1	1	83 (R)	−0.0006
			Sandy Hook, N. J.	28 900	1	1	66	−0.0021
			New York, N. Y.	27 500	1	1	63	−0.0038
			Altoona, Pa.	28 600	1	2	80 (R)	−0.0015
			State College, Pa.	28 400	3	1	82 (R)	−0.0007
		Indoors	Coco Solo Islands, C. Z.	31 700	1	1	82 (R)	−0.0003
			New Kensington, Pa.	32 400	1	1	69	−0.0005
			Cambridge, Mass.	32 000	0.2 (N)	1	108	−0.0003
			Tucson, Ariz.	30 100	0.2 (N)	1	91	−0.0005

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[a] All specimens exposed at Rochester, N. Y., were missing.

[b] The expansion values on the outdoor exposure specimens did not detect whether the bars expanded or contracted upon aging because the specimens were noticeably eroded. Therefore, the values are a measure of the extent of erosion combined with the aging change.

(R)—Rockwell hardness, E Scale (⅛-in. ball, 100-kg. load).

(N)—0.050-in. deep V-notch, having a base radius of 0.010 in., was machined in bars 3 in. in length cut from the center of 6-in. bars.

TABLE III.—MECHANICAL PROPERTIES AND EXPANSION DATA ON MAGNESIUM-BASE DIE-CASTING ALLOY SPECIMENS AFTER 5-YEAR ATMOSPHERIC EXPOSURE.—*Concluded.*

Alloy	Producer	Exposure[a]		Tensile Strength, psi.	Charpy Impact Strength, ft-lb. (¼ by ¼-in. Bars)	Elongation in 2 in., per cent	Brinell Hardness	Expansion, in. per 6 in.[b]
		Type	Location					
No. 313	H	Outdoors	Key West, Fla.	28 800	1	2	75 (R)	−0.0005
			Sandy Hook, N. J.	27 700	1	2	57	−0.0014
			New York, N. Y.	27 300	1	2	57	−0.0024
			Altoona, Pa.	25 300	1	2	65 (R)	−0.0014
			State College, Pa.	28 700	1	2	72 (R)	−0.0008
		Indoors	Coco Solo Islands, C. Z.	29 800	1	3	69 (R)	−0.0001
			New Kensington, Pa.	30 200	1	2	60	−0.0004
			Cambridge, Mass.	29 200	0.3 (N)	1	72	−0.0004
			Tucson, Ariz.	29 400	0.3 (N)	1	82	−0.0007
No. 313	K	Outdoors	Key West, Fla.	32 700	2	3	74 (R)	−0.0002
			Sandy Hook, N. J.	29 500	2	3	57	−0.0008
			New York, N. Y.	30 300	2	4	58	−0.0026
			Altoona, Pa.	28 900	2	4	70 (R)	−0.0013
			State College, Pa.	31 400	2	3	73 (R)	−0.0005
		Indoors	Coco Solo Islands, C. Z.	32 000	2	4	78 (R)	0.0004
			New Kensington, Pa.	32 600	2	3	60	0.0000
			Cambridge, Mass.	32 500	0.4 (N)	3	68	−0.0001
			Tucson, Ariz.	29 500	0.3 (N)	2	79	−0.0003
No. 313	M	Outdoors	Key West, Fla.	29 900	2	3	77 (R)	−0.0005
			Sandy Hook, N. J.	26 800	1	2	59	−0.0019
			New York, N. Y.	24 600	2	2	58	−0.0033
			Altoona, Pa.	25 300	2	3	69 (R)	−0.0020
			State College, Pa.	26 100	1	1	72 (R)	−0.0008
		Indoors	Coco Solo Islands, C. Z.	29 000	2	2	75 (R)	−0.0004
			New Kensington, Pa.	28 600	2	2	60	−0.0004
			Cambridge, Mass.	29 200	0.3 (N)	2	71	−0.0004
			Tucson, Ariz.	29 500	0.3 (N)	2	79	−0.0003
No. 314	H	Outdoors	Key West, Fla.	29 600	2	2	75 (R)	−0.0006
			Sandy Hook, N. J.	28 100	2	3	56	−0.0015
			New York, N. Y.	23 500	2	2	56	−0.0020
			Altoona, Pa.	25 500	2	4	69 (R)	−0.0019
			State College, Pa.	29 200	2	2	72 (R)	−0.0008
		Indoors	Coco Solo Islands, C. Z.	28 100	1	2	73 (R)	−0.0003
			New Kensington, Pa.	29 600	2	2	59	−0.0003
			Cambridge, Mass.	31 500	0.4 (N)	3	88	−0.0003
			Tucson, Ariz.	32 200	0.4 (N)	3	80	−0.0003
No. 314	K	Outdoors	Key West, Fla.	32 600	3	5	69 (R)	0.0001
			Sandy Hook, N. J.	28 900	3	5	53	−0.0006
			New York, N. Y.	25 200	3	4	54	−0.0013
			Altoona, Pa.	26 400	2	3	67 (R)	−0.0019
			State College, Pa.	33 000	2	5	68 (R)	−0.0004
		Indoors	Coco Solo Islands, C. Z.	35 000	3	6	74 (R)	0.0001
			New Kensington, Pa.	34 000	4	6	56	−0.0001
			Cambridge, Mass.	35 600	0.5 (N)	6	85	0.0000
			Tucson, Ariz.	33 200	0.5 (N)	4	74	−0.0001
No. 314	M	Outdoors	Key West, Fla.	26 200	2	1	75 (R)	−0.0004
			Sandy Hook, N. J.	26 100	2	2	56	−0.0015
			New York, N. Y.	23 300	1	3	55	−0.0032
			Altoona, Pa.	25 300	2	3	66 (R)	−0.0018
			State College, Pa.	27 700	2	3	66 (R)	−0.0005
		Indoors	Coco Solo Islands, C. Z.	35 000	3	6	74 (R)	0.0001
			New Kensington, Pa.	34 000	4	6	56	−0.0001
			Cambridge, Mass.	29 200	0.4 (N)	2	91	−0.0002
			Tucson, Ariz.	26 900	0.4 (N)	1	80	−0.0001

[a] All specimens exposed at Rochester, N. Y., were missing.

[b] The expansion values on the outdoor exposure specimens did not detect whether the bars expanded or contracted upon aging because the specimens were noticeably eroded. Therefore, the values are a measure of the extent of erosion combined with the aging change.

(R)—Rockwell hardness, E Scale (⅛-in. ball, 100-kg. load).

(N)—0.050-in. deep V-notch, having a base radius of 0.010 in., was machined in bars 3 in. in length cut from the center of 6-in. bars.

TABLE IV.—MECHANICAL PROPERTIES AND EXPANSION DATA ON ZINC-BASE DIE-CASTING ALLOY SPECIMENS AFTER 5-YEAR ATMOSPHERIC EXPOSURE.

Alloy	Producer	Exposure[a]		Tensile Strength, psi.	Charpy Impact Strength, ft-lb. (¼ by ¼-in. Bars)	Elongation in 2 in., per cent	Brinell Hardness	Expansion, in. per 6 in.
		Type	Location					
No. XXIa[b]	A	Outdoors	Key West, Fla.	49 700	4	4	98	0.0052
			Sandy Hook, N. J.	48 600	13	4	92 (R)	0.0011
			New York, N. Y.	47 300	15	6	93 (R)	0.0015
			Altoona, Pa.	46 300	16	7	95 (R)	0.0011
			State College, Pa.	47 100	15	5	99 (R)	0.0008
		Indoors	Coco Solo Islands, C. Z.	49 600	4	7	98	0.0059
			New Kensington, Pa.	50 300	15	9	97 (R)	0.0025
			Cambridge, Mass.	50 400	9	7	96	0.0025
			Tucson, Ariz.	51 100	6	4	95 (R)	0.0047
No. XXIa	D	Outdoors	Key West, Fla.	48 900	3	3	99	0.0060
			Sandy Hook, N. J.	46 500	12	2	96 (R)	0.0016
			New York, N. Y.	47 800	12	3	96 (R)	0.0020
			Altoona, Pa.	47 800	17	4	98 (R)	0.0016
			State College, Pa.	49 100	9	3	100 (R)	0.0015
		Indoors	Coco Solo Islands, C. Z.	48 400	4	2	100	0.0072
			New Kensington, Pa.	50 800	14	7	98 (R)	0.0029
			Cambridge, Mass.	49 500	14	4	99	0.0030
			Tucson, Ariz.	50 700	6	3	97 (R)	0.0058
No. XXIa	J	Outdoors	Key West, Fla.	50 500	3	4	96	0.0026
			Sandy Hook, N. J.	48 800	16	5	93 (R)	−0.0006
			New York, N. Y.	47 000	19	5	95 (R)	−0.0006
			Altoona, Pa.	45 600	17	6	95 (R)	−0.0002
			State College, Pa.	47 200	17	5	99 (R)	−0.0008
		Indoors	Coco Solo Islands, C. Z.	49 900	5	4	100	0.0035
			New Kensington, Pa.	50 200	18	11	96 (R)	0.0001
			Cambridge, Mass.	50 100	16	6	102	0.0004
			Tucson, Ariz.	51 700	9	3	96 (R)	0.0021
No. XXIa	P	Outdoors	Key West, Fla.	46 000	10	6	96	0.0019
			Sandy Hook, N. J.	44 900	17	7	92 (R)	−0.0003
			New York, N. Y.	43 800	23	8	93 (R)	−0.0005
			Altoona, Pa.	42 100	20	8	95 (R)	−0.0003
			State College, Pa.	43 900	20	6	97 (R)	−0.0009
		Indoors	Coco Solo Islands, C. Z.	47 100	7	5	98	0.0033
			New Kensington, Pa.	44 600	22	10	96 (R)	0.0002
			Cambridge, Mass.	46 200	13	6	93	0.0002
			Tucson, Ariz.	46 000	13	4	94 (R)	0.0010
No. XXIa	S	Outdoors	Key West, Fla.	53 500	1	3	100	0.0092
			Sandy Hook, N. J.	57 800	3	3	93 (R)	0.0028
			New York, N. Y.	55 200	4	3	99 (R)	0.0034
			Altoona, Pa.	55 200	4	6	100 (R)	0.0026
			State College, Pa.	57 200	5	3	102 (R)	0.0024
		Indoors	Coco Solo Islands, C. Z.	53 400	2	3	102	0.0103
			New Kensington, Pa.	57 600	8	10	100 (R)	0.0056
			Cambridge, Mass.	56 800	6	6	106	0.0060
			Tucson, Ariz.	55 600	2	4	97 (R)	0.0099
No. XXIa	Z	Outdoors	Key West, Fla.	53 700	5	5	98	0.0060
			Sandy Hook, N. J.	51 500	14	7	96 (R)	0.0012
			New York, N. Y.	49 700	15	5	98 (R)	0.0017
			Altoona, Pa.	48 900	21	9	98 (R)	0.0009
			State College, Pa.	50 300	17	6	101 (R)	0.0008
		Indoors	Coco Solo Islands, C. Z.	53 600	4	5	104	0.0072
			New Kensington, Pa.	53 100	20	9	99 (R)	0.0023
			Cambridge, Mass.	53 700	16	7	104	0.0027
			Tucson, Ariz.	55 000	13	6	98 (R)	0.0056

[a] All specimens exposed at Rochester, N. Y., were missing.
[b] These alloy XXI specimens were marked with an "a" to differentiate them from those exposed in 1929.
(R)—Rockwell hardness, E Scale (⅛-in. ball, 100-kg. load).

TABLE IV.—MECHANICAL PROPERTIES AND EXPANSION DATA ON ZINC-BASE DIE-CASTING ALLOY SPECIMENS AFTER 5-YEAR ATMOSPHERIC EXPOSURE.—*Continued.*

Alloy	Producer	Exposure[a]		Tensile Strength, psi.	Charpy Impact Strength, ft-lb. (¼ by ¼-in. Bars)	Elongation in 2 in., per cent	Brinell Hardness	Expansion, in. per 6 in.
		Type	Location					
No. XXIII	A	**Outdoors**	Key West, Fla.	35 000	33	20	64	0.0000
			Sandy Hook, N. J.	35 900	42	10	76 (R)	−0.0004
			New York, N. Y.	34 100	38	7	77 (R)	−0.0006
			Altoona, Pa.	33 600	29	19	79 (R)	−0.0005
			State College, Pa.	35 500	39	15	82 (R)	−0.0004
		Indoors	Coco Solo Islands, C. Z.	33 800	26	19	61	−0.0002
			New Kensington, Pa.	34 900	27	19	80 (R)	−0.0004
			Cambridge, Mass.	34 700	33	19	65	−0.0004
			Tucson, Ariz.	36 100	43	16	76 (R)	−0.0006
No. XXIII	J	Outdoors	Key West, Fla.	34 400	38	17	60	−0.0001
			Sandy Hook, N. J.	36 000	43	7	75 (R)	−0.0005
			New York, N. Y.	34 100	41	7	77 (R)	−0.0008
			Altoona, Pa.	33 100	26	12	76 (R)	−0.0008
			State College, Pa.	35 000	37	12	82 (R)	−0.0005
		Indoors	Coco Solo Islands, C. Z.	33 600	38	16	61	−0.0003
			New Kensington, Pa.	34 300	35	19	77 (R)	−0.0005
			Cambridge, Mass.	34 500	32	12	59	−0.0005
			Tucson, Ariz.	36 000	41	12	77 (R)	−0.0007
No. XXIII	P	Outdoors	Key West, Fla.	33 600	42	27	62	0.0005
			Sandy Hook, N. J.	35 600	43	15	76 (R)	−0.0004
			New York, N. Y.	34 100	44	13	77 (R)	−0.0009
			Altoona, Pa.	32 800	28	24	76 (R)	−0.0008
			State College, Pa.	34 200	33	21	81 (R)	−0.0005
		Indoors	Coco Solo Islands, C. Z.	34 100	39	25	61	−0.0002
			New Kensington, Pa.	33 700	40	26	78 (R)	−0.0005
			Cambridge, Mass.	34 200	35	15	61	−0.0007
			Tucson, Ariz.	35 500	40	21	77 (R)	−0.0006
No. XXIII	Z	Outdoors	Key West, Fla.	34 800	42	19	65	0.0000
			Sandy Hook, N. J.	36 500	44	13	76 (R)	−0.0002
			New York, N. Y.	35 000	44	10	79 (R)	−0.0005
			Altoona, Pa.	34 000	36	18	80 (R)	−0.0005
			State College, Pa.	35 600	41	18	85 (R)	−0.0003
		Indoors	Coco Solo Islands, C. Z.	33 800	26	19	61	−0.0002
			New Kensington, Pa.	35 300	42	18	81 (R)	−0.0004
			Cambridge, Mass.	35 500	34	18	65	−0.0004
			Tucson, Ariz.	36 500	47	15	77 (R)	−0.0004
No. XXV	A	Outdoors	Key West, Fla.	39 600	34	16	76	−0.0009
			Sandy Hook, N. J.	41 600	40	6	84 (R)	−0.0009
			New York, N. Y	40 100	39	7	86 (R)	−0.0019
			Altoona, Pa.	37 300	29	6	88 (R)	−0.0013
			State College, Pa.	40 100	35	8	90 (R)	−0.0011
		Indoors	Coco Solo Islands, C. Z.	38 700	35	20	71	−0.0008
			New Kensington, Pa.	40 100	31	18	87 (R)	−0.0013
			Cambridge, Mass.	40 100	36	13	79	−0.0012
			Tucson, Ariz.	41 000	38	12	86 (R)	−0.0010
No. XXV	D	Outdoors	Key West, Fla.	40 500	40	10	78	−0.0003
			Sandy Hook, N. J.	42 600	40	1	87 (R)	−0.0008
			New York, N. Y.	39 300	42	3	87 (R)	−0.0006
			Altoona, Pa.	39 100	30	5	90 (R)	−0.0008
			State College, Pa.	42 200	37	5	93 (R)	−0.0008
		Indoors	Coco Solo Islands, C. Z.	40 100	37	10	80	−0.0005
			New Kensington, Pa.	39 700	43	6	88 (R)	−0.0010
			Cambridge, Mass.	40 800	20	10	77	−0.0009
			Tucson, Ariz.	39 900	41	7	87 (R)	−0.0008

[a] All specimens exposed at Rochester, N. Y., were missing.

(R)—Rockwell hardness, E Scale (⅛-in. ball, 100-kg. load).

TABLE IV.—MECHANICAL PROPERTIES AND EXPANSION DATA ON ZINC-BASE DIE-CASTING ALLOY SPECIMENS AFTER 5-YEAR ATMOSPHERIC EXPOSURE.—*Concluded.*

Alloy	Producer	Exposure[a]		Tensile Strength, psi.	Charpy Impact Strength, ft-lb. (¼ by ¼-in. Bars)	Elongation in 2 in., per cent	Brinell Hardness	Expansion, in. per 6 in.
		Type	Location					
No. XXV	J	Outdoors	Key West, Fla.	38 000	30	10	75	−0.0008
			Sandy Hook, N. J.	39 500	37	6	84 (R)	−0.0014
			New York, N. Y.	37 800	38	6	83 (R)	−0.0014
			Altoona, Pa.	35 700	21	6	86 (R)	−0.0015
			State College, Pa.	38 500	33	7	90 (R)	−0.0014
		Indoors	Coco Solo Islands, C. Z.	36 900	31	9	73	−0.0010
			New Kensington, Pa.	37 800	33	11	86 (R)	−0.0015
			Cambridge, Mass.	38 500	25	8	71	−0.0015
			Tucson, Ariz.	39 200	35	6	83 (R)	−0.0013
No. XXV	P	Outdoors	Key West, Fla.	38 300	34	12	76	−0.0013
			Sandy Hook, N. J.	40 000	39	10	82 (R)	−0.0023
			New York, N. Y.	38 100	35	9	82 (R)	−0.0019
			Altoona, Pa.	36 200	28	9	86 (R)	−0.0018
			State College, Pa.	39 100	33	8	90 (R)	−0.0020
		Indoors	Coco Solo Islands, C. Z.	38 300	30	13	76	−0.0020
			New Kensington, Pa.	39 000	38	17	86 (R)	−0.0022
			Cambridge, Mass.	39 000	33	12	71	−0.0022
			Tucson, Ariz.	39 800	41	15	84 (R)	−0.0022
No. XXV	S	Outdoors	Key West, Fla.	40 600	25	15	78	−0.0003
			Sandy Hook, N. J.	43 500	37	11	83 (R)	−0.0009
			New York, N. Y.	41 400	34	8	85 (R)	−0.0006
			Altoona, Pa.	39 600	24	9	89 (R)	−0.0009
			State College, Pa.	42 300	32	10	92 (R)	−0.0007
		Indoors	Coco Solo Islands, C. Z.	39 800	29	11	76	−0.0007
			New Kensington, Pa.	42 000	32	20	88 (R)	−0.0007
			Cambridge, Mass.	41 000	35	10	75	−0.0009
			Tucson, Ariz.	41 900	32	16	83 (R)	−0.0007
No. XXV	Z	Outdoors	Key West, Fla.	38 600	39	15	80	−0.0007
			Sandy Hook, N. J.	40 900	45	8	86 (R)	−0.0010
			New York, N. Y.	39 800	41	10	85 (R)	−0.0009
			Altoona, Pa.	37 300	33	8	89 (R)	−0.0011
			State College, Pa.	40 200	41	9	93 (R)	−0.0010
		Indoors	Coco Solo Islands, C. Z.	38 200	40	21	76	−0.0006
			New Kensington, Pa.	39 200	44	18	88 (R)	−0.0011
			Cambridge, Mass.	39 700	37	13	78	−0.0010
			Tucson, Ariz.	39 900	44	17	86 (R)	−0.0009

[a] All specimens exposed at Rochester, N. Y., were missing.
(R) —Rockwell hardness, E Scale (⅛-in. ball, 100-kg. load).

REPORT OF COMMITTEE B-7*

ON

LIGHT METALS AND ALLOYS, CAST AND WROUGHT

Committee B-7 on Light Metals and Alloys, Cast and Wrought, was not very active during the first half of the past year because of the continued restrictions on meetings and travel and the necessary activities of its members incidental to the early reconversion period. The committee met on March 1, 1946, in Pittsburgh, Pa., however, and heard very interesting reports on the current and proposed activities of its subcommittees, as covered later in this report, and authorized considerable activity with respect to specifications. The committee, through its chairman, also is attempting to work out, with Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys, a program of corrosion of tests of light metals and alloys.

Changes in membership during the past year have increased the total to 66; of whom 29 are producers, 24 consumers, and 13 general interest members.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, I. V. Williams.

Vice-Chairman, Walter Bonsack.

Secretary, J. J. Bowman.

The committee in its 1944 report[1] included a description of the code system for the designation of magnesium-base alloys which had been adopted by the committee and is now used in the specifications for these alloys under the jurisdiction of Committee B-7. Experience has indicated that slight modifications in the method of applying the system are desirable. The following new method for applying the system will therefore be used in the future:

Code System for Designation of Magnesium Alloys.—The letters designating the alloying constituents will be arranged in order from the element present in the greatest amount to that present in the least amount. Numbers indicating the nominal amount of each indicated element will follow the letters and be arranged in the same order. When a range is specified for the element, the nominal percentage will be the mean of the range, rounded off to the nearest whole number using the methods described in the Tentative Recommended Practices for Designation of Numerical Requirements in Standards (E 29 - 40 T). If only a minimum percentage for the element is specified, the minimum will be rounded off to the nearest whole number and this rounded-off minimum considered as the nominal percentage. Small amounts of manganese are present, and essential, in all of the alloys but will not be indicated in the designation; the presence of manganese in small amounts being implied in all designations. When only a maximum is specified for an element, it is considered an impurity and is ignored in the designation. The alloy constituents will be designated by the following letters:

A—aluminum
M—manganese[a]
S—silicon
Z—zinc
T—tin
D—cadmium
E—cepium
K—zirconium
X—high purity[b]

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 44, p. 266 (1944).

[a] To be used only where manganese is a primary constituent.

[b] To be used where the impurities iron and nickel are controlled to low limits.

The changes will affect the designations of only three alloys in current tentative specifications: Alloys AZ90 and AZ90X in Specifications B 93 and B 94 become AZ91 and AZ91X, respectively, and alloy AT35 in Specifications B 91 becomes TA54.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is submitting three new tentative specifications, revisions in three tentatives, revision of one standard for immediate adoption, and the withdrawal of five tentatives. The committee also has under consideration revisions in a number of other specifications and work is actively under way on the preparation of a number of new tentative specifications. Much of this work has not been completed in time for inclusion in this report and these recommendations will be submitted at a later date to the Society through the Administrative Committee on Standards.

I. NEW TENTATIVES

The committee recommends the following three new specifications for publication as tentative as appended hereto[2]:

Tentative Specifications for:

Aluminum-Alloy Sheets and Plates,
Aluminum-Alloy Drawn Seamless Tubing, and
Aluminum-Alloy Bars, Rods and Wire.

II. REVISION OF TENTATIVES

The committee recommends that the Tentative Specifications for Magnesium-Base Alloy Sheet (B 90 – 45 T)[3] be revised as follows:

Section 9 (c).—Delete the following sentence: "In case an exact length is specified, the permissible variation in length shall be plus $\frac{1}{8}$ in. with no minus variation."

This statement is inconsistent with the requirements of the table of length tolerances and does not fully conform with commercial practice.

The committee also recommends that the Tentative Specifications for Aluminum-Manganese Alloy Sheet and Plate for Use in Welded Pressure Vessels (B 126 – 44 T)[4] and for Aluminum Sheet and Plate for Use in Welded Pressure Vessels (B 178 – 44 T)[4] be revised by deleting the present tables of thickness tolerances and substituting the accompanying Table I.

III. REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends the following revision in the Standard Specifications for Aluminum Ingots for Remelting (B 24 – 44)[4] for immediate adoption and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that this recommendation may be referred to letter ballot of the Society.

Scope and Table I.—Delete the present four grades in the scope and Table I, substituting grades 99+ and 99.75 for which the composition requirements shall be changed to read as follows:

Elements	Grade 99+	Grade 99.75
Copper, max., per cent	0.10	0.05
Iron, max., per cent	0.50	0.20
Silicon, max., per cent	0.30	0.10
Magnesium, max., per cent	0.05	0.03
Others, each, max., per cent	0.05	0.03
Total of all impurities, max., per cent	0.70	0.25
Aluminum (by difference), min., per cent	99.30	99.75

IV. WITHDRAWAL OF TENTATIVES

The committee recommends withdrawal of the following five tentative specifications:

Tentative Specifications for:

Aluminum-Alloy (Duralumin) Sheet and Plate (Aluminum - Copper - Magnesium - Manganese) (B 78 – 44 T),

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[2] These specifications were accepted as tentative by the Society and appear in the 1946 Book of A.S.T.M. Standards, Part I-B.

[3] 1945 Supplement to Book of A.S.T.M. Standards, Part I.

[4] 1944 Book of A.S.T.M. Standards, Part I.

Aluminum Sheet and Plate (B 25 – 44 T),
Aluminum-Manganese Alloy Sheet and Plate (B 79 – 44 T),
Aluminum-Alloy (Duralumin) Bars, Rods, Wire, and Shapes (B 89 – 44 T), and
Aluminum-Magnesium-Chromium Alloy Sheet and Plate (B 109 – 44 T).

The above specifications are being discontinued because the material covered by Specifications B 78 is no longer commercially available and the aluminum-alloy products covered by the remaining four specifications are being provided for in the new tentatives submitted herewith.

TABLE I.—PERMISSIBLE VARIATIONS IN THICKNESS OF FLAT AND COILED SHEETS AND PLATES, PLUS OR MINUS, IN.

Thickness, in.	Width						
	18 in. and under	Over 18 to 36 in.	Over 36 to 54 in.	Over 54 to 72 in.	Over 72 to 90 in.	Over 90 to 102 in.	Over 102 to 132 in.
3.000 to 2.751	0.090	0.090	0.090	0.090	0.120	0.150	.....
2.750 to 2.251	0.075	0.075	0.075	0.075	0.100	0.125	0.125
2.250 to 1.876	0.060	0.060	0.060	0.060	0.080	0.100	0.100
1.875 to 1.626	0.052	0.052	0.052	0.052	0.070	0.088	0.088
1.625 to 1.376	0.045	0.045	0.045	0.045	0.060	0.075	0.075
1.375 to 1.126	0.040	0.040	0.040	0.040	0.052	0.065	0.065
1.125 to 0.876	0.035	0.035	0.035	0.035	0.045	0.055	0.055
0.875 to 0.626	0.030	0.030	0.030	0.030	0.037	0.045	0.045
0.625 to 0.439	0.025	0.025	0.025	0.025	0.030	0.035	0.035
0.438 to 0.321	0.019	0.019	0.019	0.019	0.023	0.026	0.026
0.320 to 0.250	0.013	0.013	0.013	0.015	0.017	0.020	.....
0.249 to 0.204	0.009	0.009	0.011	0.013	0.015	0.017	.....
0.203 to 0.173	0.007	0.007	0.009	0.011	0.013	0.015	.....
0.172 to 0.141	0.006	0.006	0.008	0.009	0.011	0.012	.....
0.140 to 0.109	0.0045	0.0045	0.005	0.007	0.009	0.010	.....
0.108 to 0.097	0.0035	0.004	0.005	0.007	0.009	0.010	.....
0.096 to 0.077	0.003	0.003	0.004	0.006	0.008	.....	.....
0.076 to 0.069	0.0025	0.003	0.004	0.006	0.008	.....	.....
0.068 to 0.046	0.0025	0.003	0.004	0.005	0.007	.....	.....
0.045 to 0.037	0.002	0.0025	0.003	0.004	.....	.....	.....
0.036 to 0.029	0.002	0.002	0.0025	0.0035	.....	.....	.....
0.028 to 0.018	0.0015	0.002	0.0025	.....	.....	.....	.....
0.017 to 0.011	0.0015	0.0015	0.002	.....	.....	.....	.....
0.010 to 0.008	0.001	0.0015	.....	.....	.....	.....	.....
0.007 to 0.006	0.001	0.001	.....	.....	.....	.....	.....

V. TENTATIVES CONTINUED AS TENTATIVE WITHOUT REVISION

Although revisions of many of these tentatives are in process, the work is incomplete and the committee finds it necessary at this time to recommend that the following specifications be continued as tentative without revision:

Tentative Specifications for:

Aluminum-Base Alloy Sand Castings (B 26 – 44 T),
Aluminum for Use in Iron and Steel Manufacture (B 37 – 42 T),
Aluminum-Base Alloys in Ingot Form for Sand Castings (B 58 – 44 T),
Aluminum-Base Alloy Permanent Mold Castings (B 108 – 44 T),
Aluminum-Base Alloys in Ingot Form for Permanent Mold Castings (B 112 – 44 T), and
Aluminum-Base Alloys in Ingot Form for Die Castings (B 125 – 44 T).

These recommendations in this report have been submitted to letter ballot of the committee, which consists of 66 members, with the results shown in Table II.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum and Aluminum-Alloy Ingots (A. Sugar, chairman).—The activities of this subcommittee with respect to specifications are largely contingent upon the work of Subcommittee II of Committee B-7 and Subcommittee I of Committee B-6 on Die-Cast Metals and Alloys. The subcommittee is maintaining close association with the work of these latter groups in order that the specifications for alloy ingots may be kept consistent with those for the finished castings. In addition, the subcommittee has appointed a small task group to consult with the steel industry for the purpose of determining whether a revision of the Tentative Specifications for Aluminum for Use in Iron and Steel Manufacture (B 37 – 42 T) is desirable.

TABLE II.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVES			
Spec. for Aluminum-Alloy Sheets and Plates	25	1	13
Spec. for Aluminum-Alloy Drawn Seamless Tubing	26	0	13
Spec. for Aluminum-Alloy Bars, Rods and Wire	26	0	13
II. REVISIONS OF TENTATIVES			
Spec. for Magnesium-Base Alloy Sheet (B 90 – 45 T)	25	1	13
Spec. for Aluminum-Manganese Alloy Sheet and Plate for Use in Welded Pressure Vessels (B 126 – 44 T)	23	0	16
Spec. for Aluminum Sheet and Plate for Use in Welded Pressure Vessels (B 178 – 44 T)	23	0	16
III. REVISION OF STANDARD, IMMEDIATE ADOPTION			
Spec. for Aluminum Ingots for Remelting (B 24 – 44)	29	0	10
IV. WITHDRAWAL OF TENTATIVES			
Spec. for Aluminum-Alloy (Duralumin) Sheet and Plate (B 78 – 44 T)	27	0	12
Spec. for Aluminum Sheet and Plate (B 25 – 44 T)	27	0	12
Spec. for Aluminum-Manganese Alloy Sheet and Plate (B 79 – 44 T)	26	0	13
Spec. for Aluminum-Alloy (Duralumin) Bars, Rods, Wire and Shapes (B 89 – 44 T)	27	0	12
Spec. for Aluminum-Magnesium-Chromium Alloy Sheet and Plate (B 109 – 44 T)	26	0	13

After a review of the present commercial standards, the subcommittee has recommended, for immediate adoption, the changes in the Standard Specifications for Aluminum Alloy Ingots for Remelting (B 24 – 44) described earlier in this report.

Subcommittee II on Aluminum-Alloy Castings (I. V. Williams, chairman).—Discussion of the two tentatives for aluminum-alloy castings (B 26 and B 58) at the last meeting of this subcommittee led to the conclusion that they should be thoroughly reviewed. Accordingly, task groups were appointed to make these reviews and were directed to report their conclusions as promptly as possible.

Subcommittee III on Wrought Aluminum and Wrought Aluminum Alloys (P. V. Faragher, chairman).—Since the A.S.T.M. specifications for wrought aluminum alloys do not offer a reasonably complete coverage of the materials available commercially, this subcommittee is preparing a number of new specifications. Several of these specifications have been completed and are recommended for publication as tentative; the remainder will be completed very shortly. Incidental to this review, it was concluded that the Tentative Specifications for Aluminum-Alloy (Duralumin) Sheet and Plate (B 78 – 44 T) was no longer necessary, since the material it describes has been almost completely superseded in commercial applications by other materials. Four other tentatives for wrought products are made obsolete by the proposed new specifications. As set forth elsewhere in this report, the withdrawal of these tentatives is being recommended.

Subcommittee IV on Magnesium and Magnesium Alloys, Cast and Wrought (H. W. Schmidt, chairman).—The specifications under the jurisdiction of this subcommittee were completely reviewed last year and have not yet stood in their present form long enough to warrant further changes. A minor change in the Tentative Specifications for Magnesium-Base Alloy Sheet (B 90 – 45 T), overlooked last year, is being made at this time. This subcommittee is preparing new tentative specifications for magnesium-alloy tubing, which will round out the Society's coverage of the wrought magnesium-alloy products.

Subcommittee V on Testing of Light Alloys (R. L. Templin, chairman) met in Pittsburgh and reviewed those activities of Committee E-1 on Methods of Testing having a bearing on the specifications under the jurisdiction of Committee B-7. Since, however, these activities had not yet resulted in formal recommendations to the Society, Subcommittee V could do nothing beyond discussing the work in progress and its possible application to the testing of light metals.

The subcommittee also heard an informal report of some work being done to determine the limits of error to be expected in the determination of elongation in specimens having a typical elongation of about 0.5 per cent in 2 in. A technical paper describing the work and the results obtained will be submitted to the Society as soon as the investigation is completed.

Subcommittee VI on Anodic Oxidation of Aluminum and Aluminum Alloys (J. D. Edwards, chairman) has been inactive during the past year and does not, at the moment, have any program for future work. Associates of the chairman have been investigating a recently developed instrument for nondestructively measuring the thickness of oxide coatings on aluminum and have offered to make their findings available to the subcommittee at an early date. Further tests of the instrument will be conducted by the subcommittee if these preliminary data appear to warrant them.

Subcommittee VII on Codification of Light Alloys (I. V. Williams, chairman). —As pointed out earlier in this report, certain difficulties in applying the proposed code system for magnesium alloys have been encountered and a minor change in the system has been proposed. Some adverse comment on the present code system for aluminum alloys also has been received and discussions are in progress to determine whether acceptable modifications can be worked out. In addition, the subcommittee has been asked to develop, if possible, a code system to designate the commonly used heat treatments for both aluminum and magnesium alloys and is in the midst of a study of this problem.

This report has been submitted to a letter ballot of the committee, which consists of 66 members; 39 members returned their ballots, of whom 33 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

D. L. COLWELL,
Chairman.

J. J. BOWMAN,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-7 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:

Magnesium-Base Alloy Extruded Round Tubing (B 217 - 46 T), and
Aluminum-Base Alloys in Ingot Form for Use in the Manufacture of Castings (B 179 - 46 T).

Revision of Tentative Specifications for:

Aluminum and Aluminum-Alloy Sheet and Plate (B 209 - 46 T),
Magnesium-Base Alloy Sheet (B 90 - 46 T),
Aluminum for Use in Iron and Steel Manufacture (B 37 - 46 T),
Aluminum-Base Alloy Sand Castings (B 26 - 46 T), and
Aluminum-Base Alloy Permanent Mold Castings (B 108 - 46 T).

The new specifications B 217 were accepted as tentative on September 9, 1946. The other recommendations were accepted by the Standards Committee on November 6, 1946. The new and revised tentatives appear in the 1946 Book of A.S.T.M. Standards, Part I-B.

At the same time the Standards Committee accepted the recommendation of Committee B-7 that the following tentatives be withdrawn:

Tentative Specifications for:

Aluminum-Base Alloys in Ingot Form for Sand Castings (B 58 - 44 T),
Aluminum-Base Alloys in Ingot Form for Permanent Mold Castings (B 112 - 44 T), and
Aluminum-Base Alloys in Ingot Form for Die Castings (B 125 - 44 T).

REPORT OF COMMITTEE B-8*

ON

ELECTRODEPOSITED METALLIC COATINGS

Due to restrictions on traveling and the shortage of hotel accommodations, Committee B-8 on Electrodeposited Metallic Coatings held no meetings during 1945. In lieu of committee meetings, the Advisory Committee met in Pittsburgh, Pa., on March 2, 1945, to transact the necessary committee business and prepare the 1945 report which was subsequently presented to the Society in New York, N. Y., on June 27, 1945.

The membership of the committee now totals 89, of whom 36 are classified as producers, 11 as consumers, 41 as general interest members and one as consulting member.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, R. J. McKay.
Vice-Chairman, K. G. Soderberg.
Secretary, C. H. Sample.

RECOMMENDATIONS AFFECTING STANDARDS

The committee recommends that the standard and tentatives under its jurisdiction be continued in their present status without revision.

The above recommendation has been submitted to letter ballot of the committee, which consists of 74 voting members; 53 members returned their ballots, of whom 53 have voted affirmatively, 0 negatively, and 0 marked their ballots "not voting."

ACTIVITIES OF SUBCOMMITTEES

Due to the fact that the majority of the committee members were occupied with urgent war activities, the subcommittees, with the exception of Subcommittee II, were relatively inactive during the past year. Such business as was required was carried on through correspondence by the subcommittee chairmen.

Subcommittee II on Performance Tests (H. A. Pray, chairman) has continued its atmospheric exposure tests of electrodeposited lead coatings on steel. The results of inspections to date are summarized in Table I.

The subcommittee has also started outdoor exposure tests at New York, N. Y., State College, Pa., Pittsburgh, Pa., and Kure Beach, N. C., comparing the relative protective value of various copper-nickel-chromium coatings on high-carbon steel.

This report has been submitted to letter ballot of the committee, which consists of 74 voting members; 53 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. J. McKAY,
Chairman.

C. H. SAMPLE,
Secretary.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

TABLE I.—SUMMARY OF INSPECTION DATA ON THE ATMOSPHERIC EXPOSURE TESTS OF ELECTROPLATED LEAD COATINGS.

Coating Group	Nominal Coating Thickness, in.	Number of Panels Averaged, Each Station	New York, N. Y.				State College, Pa.			
			Upper Surface		Under Surface		Upper Surface		Under Surface	
			0.87 yr.	1.37 yr.	0.87 yr.	1.37 yr.	0.78 yr.	1.26 yr.	0.78 yr.	1.26 yr.
Sulfamate lead on steel	0.00050	5	....	5P	MP-30S-1R	MP-40S	MP-40S	MP-20S	....	1P
	0.00100	5	....	1P	MP-1S	MP-2S	....	....	....	....
Fluoborate lead on steel	0.00008	3	MP-100S-20R	MP-100S-20R	MP-100S-5R	MP-100S-5R	MP-100S-60R	100R	MP-93S-7R	MP-100S-65R
	0.00025	5	MP-100S-<1R	MP-100S	MP-90S-2R	MP-100S-2R	MP-100S-50R	100R	MP-50S-1R	MP-95S-20R
	0.00050	5	MP-75S	75S	MP-40S-1R	MP-40S	MP-45S	MP-30S	5P	5P
	0.00075	5	MP-45S	60S	MP-40S-1R	MP-40S	13P	15P	1P	....
	0.00100	5	MP-2S	10S	MP-30S	MP-25S	2P	....	<1S	....
	0.00200	17	....	....	....	5P	....	....	....	....
Sulfamate lead copper-plated steel	0.00050	5	....	5P	MP-20S	MP-10S	MP-15S	MP-10S	5P	2P
	0.00100	5	....	1P	....	....	....	....	....	....
Fluoborate lead on copper-plated steel	0.00008	3	MP-100S-75R	100S-95R	MP-80S-3R	MP-85S-5R	100R	100R	100R	100R
	0.00025	5	MP-8S-1R	MP-3S	MP-15S-1R	MP-25S	MP-<1S	5P	10P	MP-2S-<1R
	0.00050	5	....	2P	MP-15S-1R	MP-10S	6P	....	....	6P
	0.00075	5	....	....	MP-3S	MP-3S	1P	....	....	....
	0.00100	5	....	....	1S	5P	....	....	....	....
	0.00200	17	....	....	....	1P	....	....	2P	....
Sulfamate lead on sheet copper	0.00025	3	....	....	....	....	2P	....	1P	....
	0.00050	3	....	....	....	....	....	....	....	....
Fluoborate lead on sheet copper	0.00025	3	....	....	....	....	....	....	....	....
	0.00050	3	....	....	....	....	....	....	....	....
Sulfamate lead-tin on steel:										
2 per cent tin	0.00100	3	....	....	MP-3S	5P	MP-100S-60R	MP-100S-25R	MP-10S-1R	MP-15S-1R
10 per cent tin	0.00100	3	....	....	....	....	MP-45S-7R	MP-50S-6R	....	1P
Commercial hot-dipped terne (20 per cent tin)	0.00017	3	MP-100S-7R	MP-100S-5R	MP-15S	MP-25S	MP-100-S70R	100R	MP-95S-20R	MP-100S-90R
Commercial hot-dipped lead (2.5 per cent tin, 2 per cent antimony)	0.00040	3	MP-60S-3R	MP-85S	MP-15S-5R	MP-40S	MP-100S-75R	MP-100S-60R	MP-98S-30R	....
Electroplated zinc on steel	0.00020	3	100R	100R	20S-30R	100R	....	....	....	....
Hot-dipped zinc on steel	0.00075	3	....	3P	....		....	....	....	....
Bare steel	....	6	100R	100R	100R	100R	100R	100R	100R	100R
Fluoborate lead on steel (damaged)	0.00050	3	....	Sl. spread	MP-10S-<1R	MP-10S	Sl. spread	Very sl. spread	1P	3P
	0.00100	3	....	Very sl. spread	MP-1S	....	....	....	....	1P
Fluoborate lead on copper-plated steel (damaged)	0.00050	3	....	Sl. spread	MP-<1S	....	....	....	1P	2P
	0.00100	3	....	Very sl. spread	....	....	....	....	....	....

TABLE I.—SUMMARY OF INSPECTION DATA ON THE ATMOSPHERIC EXPOSURE TESTS OF ELECTROPLATED LEAD COATINGS (*Concluded*).

Coating Group	Nominal Coating Thickness, in.	Number of Panels Averaged, Each Station	Kure Beach, N. C.						Tela, Honduras					
			Upper Surface			Under Surface			Upper Surface			Under Surface		
			0.81 yr.	1.34 yr.	1.83 yr.	0.81 yr.	1.34 yr.	1.83 yr.	0.83 yr.	1.33 yr.	1.83 yr.	0.83 yr.	1.33 yr.	1.83 yr.
Sulfamate lead on steel	0.00050	5	MP-75S-1R	MP-100S	100S	MP-35S	MP-50S	90S	IP-3R	5R	15R	IP	10R	15R
	0.00100	5	MP-<1S	MP-3S	MP-25S	MP-<1S	MP-<1S	5P	5P	10P	15P	....	5P	10P
Fluoborate lead or steel	0.00008	3	100R	100R	100R	100R	100R	100R	100R	100R	100R	95R	98R	100R
	0.00025	5	100S-65R	MP-100S-60R	100R	98S-3R	MP-100S-60R	100R	IP	80R	95R	IP	70R	85R
	0.00050	5	MP-95S-6R	MP-100S-1R	100S-5R	MP-45S-2R	MP-90S-3R	90S	IP	20R	30R	IP	20R	30R
	0.00075	5	MP-85S-<1R	MP-100S-1R	100S	MP-25S	MP-65S	90S	....	1P	1P	IP	5R	5R
	0.00100	5	MP-20S	MP-60S	80S	MP-6S	MP-25S	50S	IP	7P	MP	IP	MP	MP
	0.00200	17	12P	25P	MP-<1S	4P	10P	5P	3P	5P	8P	....	1P	1P
Sulfamate lead on copper-plated steel	0.00050	5	MP-55S	MP-80S	98S	MP-10S	MP-25S	65S	IP	1P	1P	IP	1P	5R
	0.00100	5	25P	MP-<1S	MP-1S	MP-<1S	MP-<1S	MP-<1S	5P	5P	10P	5P	2P	2P
Fluoborate lead on copper-plated steel	0.00008	3	100R	100R	100R	100R	100R	100R	100R	100R	100R	98R	100R	100R
	0.00025	5	MP-35S-2R	MP-80S-20R	100S-65R	MP-95S-30R	MP-100S-75R	100S-20R	IP	30R	45R	IP	35R	60R
	0.00050	5	MP-5S	MP-25S	80S	MP-12S	MP-25S	25S	IP	1P	MP	IP-2R	5R	10R
	0.00075	5	MP-10S	MP-30S	MP-60S	MP-<1S	MP-1S	MP-1S	IP-5R	8P	20P	10P	1P	MP
	0.00100	5	MP-<1S	MP-12S	MP-35S	MP-1S	MP-5S	10P-2S	10P	10P	15P	1P	7P	20P
	0.00200	17	12P	15P	MP-<1S	8P	10P	5P	5P	6P	10P	....	....	1P
Sulfamate lead on sheet copper	0.00025	3	2P	....	....	1P	....	....	....	....	....	....	....	....
	0.00050	3	....	....	....	....	....	....	....	....	....	....	....	....
Fluoborate lead on sheet copper	0.00025	3	....	....	....	....	....	....	....	....	....	....	....	....
	0.00050	3	....	....	....	....	....	....	....	....	....	....	....	....
Sulfamate lead-tin on steel:														
2 per cent tin	0.00100	3	MP-97S-3R	MP-100S	100S	MP-90S-8R	MP-90S	95S	RP-30R	80R	85R	30R	50R	55R
10 per cent tin	0.00100	3	MP-85S-<1R	MP-100S	100S	D-MP-2S-<1R	MP-30S	MP-30S	IP	40R	50R	IP	20R	40R
Commercial hot-dipped terne (20 per cent tin)	0.00017	3	100S-50R	MP-100S-80R	100R	98S-15R	MP-100S-60R	100S-20R	RP-70R	85R	90R	IP-60R	75R	80R
Commercial hot-dipped lead- (2.5 per cent tin, 2 per cent antimony)	0.00040	3	MP-95S-35R	MP-100S-45R	100S-35R	MP-90S-20R	MP-100S-30R	100S-20R	RP-40R	50R	60R	20R	30R	55R
Electroplated zinc on steel	0.00020	3	16R	50R	95R	....	....	<1R	RP	25R	40R	....	....	MP
Hot-dipped zinc on steel	0.00075	3	....	....	....	....	....	....	1P	1P	MP	....	....	....
Bare steel	....	6	100R	100R	100R	100R	100R	100R	100R	100R	100R	95R	100R	100R
Fluoborate lead on steel (damaged)	0.00050	3	Def. spread	Def. spread	Bad spread	MP-30S	MP-80S-2R	98S	....	....	....	IP	40R	60R
	0.00100	3	Def. spread	Spread	Def. spread	8P	25P	7P	....	....	....	....	8R	1P
Fluoborate lead on copper-plated steel (damaged)	0.00050	3	Sl. spread	Def. spread	Def. spread	MP-10S	MP-25S	MP-40S	....	....	....	IP	10P	5R
	0.00100	3	....	Sl. spread	Sl. spread	2P	5P	10P	....	....	....	....	7S	10S

Abbreviations and symbols used: P—number of pinholes, up to 25; MP—pinholes, more than 25; IP—pinholes, more than 25, irregularly distributed; RP—pinholes, more than 25, distributed uniformly; R—percentage of flaky rust of base metal; S—percentage of area stained, coating intact; Sl.—slight; Def.—definite.

NOTE.—Thickness of copper plate in all cases, 0.000015 in.

REPORT OF COMMITTEE B-9*

ON

METAL POWDERS AND METAL POWDER PRODUCTS

This report covers the activities of Committee B-9 on Metal Powders and Metal Powder Products for the period from June, 1944, to June, 1946. During this time, two meetings were held: in Pittsburgh, Pa., on February 28, 1945, and in Chicago, Ill., on February 27 and 28, 1946.

A special subcommittee on regulations, appointed at the February, 1944, meeting of Committee B-9, consisted of B. T. duPont, P. R. Kalischer, and A. B. Gibson. A final draft, prepared by the subcommittee, was submitted on September 30, 1944, to letter ballot of the committee which consists of 51 members; 30 members returned their ballots, of whom 28 have voted affirmatively, 0 negatively, and 2 members marked their ballots "not voting."

A temporary advisory committee consisting of F. N. Rhines, R. P. Koehring, D. O. Noel, W. R. Toeplitz, and W. A. Reich was appointed at the February, 1944, meeting of Committee B-9 and served until a permanent advisory committee was elected in December, 1944, in accordance with the new regulations to serve until the next election which was held in the spring of 1946. W. A. Reich and W. R. Toeplitz were reappointed chairman and secretary, respectively, at the February, 1945, committee meeting to serve until the next election in the spring of 1946. The advisory committee for the year of 1945 consisted of W. A. Reich, W. R. Toeplitz, R. P. Koehring, P. E. Weingart, A. Squire, F. N. Rhines and B. T. duPont.

The following slate of officers was proposed at the June, 1946, meeting of Committee B-9 by the nominating committee and unanimously elected to serve for the ensuing term of two years:

Chairman, W. A. Reich.
Vice-Chairman, B. T. duPont.
Secretary, W. R. Toeplitz.
Advisory Committee: P. E. Weingart, O. W. Ellis, M. F. Judkins, A. Squire.

RECOMMENDATIONS AFFECTING STANDARDS

The committee recommends that the following four new methods be accepted for publication as tentative, as appended hereto:[1]

Tentative Methods of:

Test for Flow Rate of Metal Powders,
Sieve Analysis of Granular Metal Powders,
Test for Apparent Density of Metal Powders, and
Sampling Finished Lots of Metal Powders.

These four recommendations have been submitted to letter ballot of the committee, which consists of 51 members, 32 members returned their ballots, of whom 27 have voted affirmatively, 0 negatively, and 5 members marked their ballots "not voting."

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Technical Data (F. N. Rhines, chairman) has been actively engaged in the formation of a glossary of terms used in powder metallurgy and at present has obtained general agreement on about 65 terms. It is expected that a rather complete set of definitions will be made made available in the next year for the use of the committee and the powder metallurgy industry. This subcommittee has been cooperating closely with the American Society for Metals in this phase of its work.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] These methods were accepted as tentative by the Society and appear in the 1946 Book of A.S.T.M. Standards, Part I-B.

Subcommittee I also is presently engaged in a program to standardize on a tension test bar for use in the field of powder metallurgy. B. T. duPont is coordinator of this work. Two questionnaires have been sent out to a representative group of about 60 metal powder producers, parts producers, fabricators, consumers, and general interests. Response to these questionnaires was very gratifying and it is expected that definite recommendations to Committee B-9 will be forthcoming before the end of 1946. This work has been closely coordinated with the Section on Tension Testing of Committee E-1 on Methods of Testing.

At the February, 1946, meeting of Committee B-9, it was decided that all standards developed by the subcommittees or sections would be approved by Subcommittee I from the standpoint of nomenclature before being submitted to committee letter ballot.

Subcommittee II on Metal Powders (D. O. Noel, chairman) prepared the four new methods mentioned earlier in the report which are being recommended for publication as tentative.

This subcommittee has three sections now working on the following problems:

Section 1 on Compressibility of Metal Powders (F. V. Lenel, chairman) is working closely with the Metal Powder Association on the establishment of a satisfactory test for compressibility.

Section 2 on Subsieve Particle Size (J. Sachse, chairman) is discussing the use of the Roller air analyzer as a standard measuring instrument for metal powders substantially over 10 microns in particle size and is also considering microscopic methods for powders under 10 microns in particle size.

Section 3 on Chemical Analysis has recognized the use so far as possible of standard A.S.T.M. methods of chemical analysis. Committee E-3 on Chemical Analysis of Metals is being asked for assistance on special methods for use in powder metallurgy covering (*1*) oxygen content or "hydrogen loss," (*2*) total iron in iron powder, and (*3*) insoluble content of metal powders.

Subcommittee II has sponsored a short article by D. O. Noel on the Hall Flowmeter which is the apparatus to be used in the apparent density and flow tests mentioned above. This article appears in the Appendix to this report.

Subcommittee III on Metal Powder Products (R. P. Koehring, chairman) is divided into five sections, whose activities are as follows:

Section 1 on Bearings (D. S. Urquhart, chairman) has been actively engaged in the preparation of specifications for oil impregnated porous bearings made from metal powders which resulted in the Tentative Specifications for Metal Powder Sintered Bearings (Oil Impregnated) (B 202 – 45 T).[2] This section is now engaged in a program aimed at developing a standard list of bearing sizes for the use of the industry. This will require a considerable amount of work on press-fit allowances and running tolerances for various types of applications in which porous bearings are used. This preliminary phase of the work is under way. Consideration is being given to the drafting of a publication on recommended applications for such bearings.

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[2] 1945 Supplement to Book of A.S.T.M. Standards, Part I.

Section 2 on Structural Parts (F. V. Lenel, chairman), has held extensive discussions on the subject of specifications for structural parts made from metal powders. In order to tackle more effectively the many problems confronting it, two study groups have been formed. A group of producers of structural parts is attempting to analyze and suggest solutions for the producer problems entering standardization. In the same way a consumer group is studying consumer problems.

At the present time, there is no standardization as to alloy compositions from which structural parts are made. The consumers of such parts have not indicated any outstanding preference for any particular composition. This makes the adoption of standard compositions a difficult problem.

The use of a separately produced test bar for acceptance purposes on strength is not generally feasible because of the variations in density within an intricate part. For this reason, acceptance tests based on service requirements on the individual part are being considered.

Section 3 on Cemented Carbides (M. F. Judkins, chairman), has three working subgroups on the following problems: (*1*) Transverse rupture test, (*2*) Hardness test, and (*3*) Analysis and grain size determination.

Other matters under consideration are methods of inspection for flaws and simulated service tests for cemented carbide parts. This section is working with the American Standards Association and other industrial groups.

Section 4 on Electrical Parts (A. B. Gibson, chairman), has two working groups; one studying magnetic test methods for pressed and sintered pole pieces and the other studying electrical contacts made from metal powders. The former group has prepared a magnetic test method for pole pieces which is being submitted to Committee B-9 for letter ballot. This method will be forwarded to Committee A-6 on Magnetic Properties for review.

The group on electrical contacts is studying the possibility of electrical resistance, hardness, and density tests, particularly for the precious metals. Because of the small sizes of such contacts it is thought that special methods will have to be evolved for these materials.

Section 5 on Friction Materials (J. R. Nurney).—It is generally felt by the members of this section that it is too early for standardization activities in the field of friction materials. The section will, however, continue as a medium for exchange of information on these materials with a view to engaging in standardizing work as soon as it becomes advisable.

This report has been submitted to letter ballot of the committee, which consists of 51 members; 27 members returned their ballots, of whom 23 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. A. REICH,
Chairman.

W. R. TOEPLITZ,
Secretary.

REPORT OF COMMITTEE C-1*

ON

CEMENT

Freed from war-time travel restrictions, Committee C-1 on Cement held two well-attended meetings during the past year: one in Washington, D. C., in October, 1945, and one in Pittsburgh, Pa., in February, 1946.

The committee lost the services of two of its long-time officers in late 1945. P. H. Bates, retiring from active work, resigned from the chairmanship of Committee C-1, a post that he had held for 16 yr. He had been an active member of the committee for a long time. The committee records with sorrow the death of Charles F. Conn in December, 1945. Mr. Conn had been a member of Committee C-1 for many years, and had long served very faithfully as a vice-chairman.

Considerable subcommittee work had been maintained throughout the last two years, and a number of recommendations affecting standards were made by the subcommittees during the past year. Some of the recommendations were submitted to the Society for interim consideration by the Administrative Committee on Standards; other recommendations are presented herewith for consideration at the Annual Meeting. Subcommittee work is being continued.

The Working Committee on Methods of Chemical Analysis (W. C. Hanna, chairman) completed a study of the Proposed Method for Determining Darex Air-Entraining Agent in Portland Cement. Seven laboratories participated in the work, as follows: California Portland Cement Co.; Central Concrete Laboratory, War Department; Dewey & Almy Chemical Co.; Lehigh Portland Cement Co.; Portland Cement Assn.; U. S. Bureau of Reclamation; Universal Atlas Cement Co. The method was revised by the subcommittee and was submitted by Committee C-1 to the Society as mentioned later in this report. The subcommittee has made some progress in planning a cooperative investigation of methods for determining the titanium oxide content of portland cement. Preliminary trials of two procedures are in progress and a survey is being made of the TiO_2 content of various portland cements. The subcommittee has cooperated with the Committee on Cement of the Federal Specifications Board in an effort to promote uniformity between the Federal and the A.S.T.M. methods of chemical analysis of cement.

The Working Committee on Strength (G. L. Lindsay, chairman) continued its studies of questions related to the methods of test for compressive strength of mortars. An extensive series of cooperative tests on six cements by six laboratories was but recently completed. The scope of that investigation is presented in Appendix I to this report. At the last meeting of Committee C-1, Clayton L. Davis, on behalf of the subcommittee, presented a detailed preliminary report on the results of the investigation. The final report will be available to the committee members at an early date.

The Sponsoring Committee on Blended Cements (R. E. Roscoe, chairman) is

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[*] Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

now engaged in the study of specifications for portland-pozzuolana cement, and is considering questions relating to portland blast-furnace slag cement for which tentative specifications were accepted by the Society during the year.

The Working Committee on Sulfate Resistance (D. G. Miller, chairman) completed a cooperative study of the lean mortar bar test as a measure of the sulfate resistance of portland cements. Six laboratories participated in the tests on ten cements. The scope of the work and the results of the tests are presented in considerable detail in Appendix II to this report. The method of making the lean mortar bar test that was used in that study is presented in Appendix III to this report. At this time the method is published as information only.

The Working Committee on the Effects of Alkalies in Portland Cement on the Durability of Concrete (R. F. Blanks, chairman) has continued its work, and recently reported to Committee C-1 the results of a cooperative study of the dependability and reproducibility of the mortar bar expansion test when incorporating the active aggregate in combination with cements of various alkali contents. The ten laboratories listed below participated in the tests of five aggregates and six cements. The total alkali contents of the cements varied from 0.16 to 1.37 per cent. The subcommittee report included the results of expansion measurements up to 52 weeks. Also presented was additional material to supplement the subcommittee's bibliography on this subject. The laboratories were as follows:

California Division of Highways, T. E. Stanton,
U. S. Bureau of Reclamation, R. F. Blanks,
California Portland Cement Co., W. C. Hanna,
Alpha Portland Cement Co., Louis Anderson,
Washington State Department of Highways, Bailey Tremper,
Central Concrete Laboratory, War Department, C. E. Wuerpel,
National Bureau of Standards, P. H. Bates,
Portland Cement Assn., F. R. McMillan,
Public Roads Administration, F. H. Jackson, and
New Jersey State Highway Dept., F. H. Baumann.

The Working Committee on Volume Change and Soundness of Portland Cement (H. F. Gonnerman, chairman) has been assembling a large amount of data from current investigations on volume change and soundness of cements. A paper on "The Influence of Gypsum on the Hydration and Properties of Portland Cement Pastes"[1] was prepared by William Lerch, and the subcommittee had been studying the effect of gypsum content on the properties of cements and the concretes made with them. Later, the available data on the effects of the SO_3 content of cement were studied jointly by this subcommittee and by the Sponsoring Committee on Portland Cement. The action resulting from that study is related later in this report.

The Sponsoring Committee on Portland Cement (C. E. Wuerpel, chairman) continued its work. Jointly, with the Working Committee on Volume Change and Soundness, it studied the question of effect of varying amounts of SO_3 content in portland cement. Later, the Sponsoring Committee proposed changes in the specification maximum limits for SO_3 content, and those changes are embodied in recommendations made by Committee C-1 elsewhere in this report. The subcommittee also submitted other proposals, leading to recommendations by Committee C-1 affecting Methods C 185 and C 183, and also Specifications C 175.

A new subcommittee was recently appointed by Committee C-1 to study the subject of SO_3 in cement, and it will endeavor to develop a method of test for the optimum SO_3 content for individual cements, which end cannot be achieved simply by a specification maximum percentage limitation. The subcommittee personnel is as follows: H. S. Meissner (chairman), H. K. Cook, W. J. McCoy, Bailey Tremper, H. F. Gonnerman, and G. L. Lindsay.

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[1] See p. 1252.

No changes are being offered at this time either in the Tentative Specifications for Masonry Cement (C 91 – 44 T), or in the Tentative Method of Test for Heat of Hydration of Portland Cement (C 186 – 44 T), and the committee recommends that those two tentatives be continued as tentative without change.

The Working Committee on Additions (H. J. Gilkey, chairman) arranged for a series of tests on a certain material that had been submitted to Committee C-1 as a grinding aid for consideration under the specification provisions governing the additions to portland cement. The tests are being made by the Cement Reference Laboratory.

The Subcommittee on Cement Reference Laboratory (G. E. Warren, chairman) continued its attention to matters relating to the work of that laboratory, especially to the question of increasing the scope of the laboratory's work. Developments in the cement and concrete testing fields gradually led to the thought that the Reference Laboratory's work should be increased to include details involved in the compression tests of concrete cylinders. The additional work would include the verification of compression testing machines up to 200,000 lb. instead of only 100,000 lb. as at present, the inspection of moist rooms, capping procedure, etc. The subcommittee reported favorably on this proposal, and Committee C-1 recommends to the Society that the work of the Cement Reference Laboratory be so enlarged. This proposed extension of the work is necessarily conditional on securing additional funds and personnel, and is also closely interwoven with the work of Committee C-9 on Concrete and Concrete Aggregates. The latter committee has approved the plan in principle, and the matter is now the subject of joint study by Committees C-9 and C-1.

The Reference Laboratory made inspections at 35 cement laboratories, practically completing the eighth inspection tour. That tour had been conducted during the war at a slower rate than usual. Considerable progress has been made on the series of tests for the Working Committee on Additions. The Reference Laboratory participated in the series of cooperative mortar tests sponsored by the Working Committee on Strength, and devoted considerable time to studying variables in miscellaneous tests. Attention was given to editorial matters relating to cement standards and the activities of Committee C-1. The Laboratory also cooperated with groups and individuals interested in the improvement and standardization of cement tests.

NEW TENTATIVES

During the year, the committee submitted three proposed tentatives for consideration by the Administrative Committee on Standards. Those three proposals were accepted for publication as tentative and were as follows:

Tentative Specifications for Portland-Blast Furnace Slag Cement (C 205 – 46 T),[2]

Tentative Method of Test for Fineness of Portland Cement by the Air-Permeability Apparatus (C 204 – 46 T),[2] and

Tentative Method for Determining Darex Air-Entraining Agent in Portland Cement[2] (to be included as part of Tentative Methods C 114 – 44 T).

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[2] 1946 Book of A.S.T.M. Standards, Part II.

The material for these three tentatives had originally been published as information in appendices to the 1944 Annual Report[3] of Committee C-1. Some revisions were made when submitting them to the Society for publication as tentative. The results of the letter ballot vote in Committee C-1 and the date of acceptance of these three recommendations by the Standards Committee are given in Table I.

I. REVISIONS OF TENTATIVES

Proposed revisions of two tentatives were also submitted to the Society during the year, and were accepted by the Standards Committee as follows:

Tentative Specifications for Air-Entraining Portland Cement (**C 175 – 46 T**).[2] The earlier tentative was modified as follows: The words restricting cement for use in pavements were deleted from the scope and title. The permissible limits on air content of the standard mortar were raised from 14 ± 4 per cent to 16 ± 4 per cent. From the section on packaging and marking there was deleted the requirement that the presence of an acceptable interground addition be indicated on the package. The committee now recommends the immediate adoption of the following further revision in C 175 – 46 T: Revise Table I by adding a footnote providing that the maximum limit for SO_3 content for Type IA Cement be 2.5 per cent when the C_3A content is greater than 8 per cent. This recommendation is based on data mentioned earlier in this report in the remarks about the Working Committee on Volume Change and Soundness, and the Sponsoring Committee on Portland Cement. The letter ballot vote on and date of acceptance of this recommendation are shown in Table I.

Tentative Method of Test for Air Content of Portland-Cement Mortar (**C 185 – 46 T**).[2]—During the year the committee submitted to the Society a recommended revision of the requirements for the Burmister mortar flow trough that is used in this method. The interim proposal was designed to promote standardization in an apparatus that is being increasingly used, and the revision was accepted by the Administrative Committee on Standards, as shown in Table I.

TABLE I.—RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS, INCLUDING RECORD OF LETTER BALLOT VOTE.[a]

Items	Affirmative	Negative	Ballots Marked "Not Voting"
New Tentative Specifications for:			
Portland-Blast Furnace Slag Cement (C 205 – 46 T), *accepted January 24, 1946*	62⅓	0	5⅓
New Tentative Methods of Test for:			
Fineness of Portland Cement by the Air-Permeability Apparatus (C 204 – 46 T), *accepted January 24, 1946*	66⅓	0	2⅓
Determining Darex Air-Entraining Agent in Portland Cement (To be included as part of C 114 – 44 T), *accepted February 6, 1946*	62⅓	0	6⅓
Revision of Tentative Specifications for:			
Air-Entraining Portland Cement (C 175 – 46 T), *accepted January 24, 1946:*			
Section on Scope	60⅓	4	4⅓
Method for Adding Resin	58⅓	1	9⅓
Air Content Limits for Mortar	59⅓	1	8⅓
Packaging and Marking	61⅓	2	5⅓
Revision of Tentative Method of Test for:			
Air Content of Portland-Cement Mortar (C 185 – 46 T), *accepted January 24, 1946*	60⅓	1	7⅓

[a] Committee had 76 net voting members during this ballot.

II. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions in the following two standards and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society:

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[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 44, p. 306 (1944).

Standard Specifications for Portland Cement (**C 150 – 44**).[4]—Earlier in this report reference was made to the data on SO_3 content of Portland cement. Based on that data, the committee recommends for immediate adoption the revision of Table I by the addition of a footnote providing that the maximum limits for SO_3 content of types I and III cements, now 2.0 and 2.5 per cent, respectively, be 2.5 and 3.0 per cent, respectively, when the C_3A content is greater than 8 per cent.

The committee also recommends the immediate revision of Section 9(*b*) by the insertion of reference to retesting in cases of local storage, the revised paragraph to read as follows:

> (*b*) Cement remaining in bulk storage at the mill prior to shipment for more than six months, or cement in bags in local storage in the hands of a vendor for more than three months, after completion of tests, may be retested before use, and may be rejected if it fails to conform to any of the requirements of these specifications.

Standard Method of Sampling Hydraulic Cement (**C 183 – 44**).[4]—Committee C-1 recommends the following revisions for immediate adoption:

Sections 3 and 4.—Combine these two sections and change to read as follows, renumbering the subsequent sections accordingly:

> 3. *Sampling.*—The cement may be sampled by any of the methods described in the following Paragraphs (*a*) to (*d*):
>
> (*a*) *From the Conveyor Delivering to Bulk Storage.*—One sample of 4 lb. or more shall be taken from at least each 2000 bags passing over the conveyor, except that the sample shall not represent more than 6 hr. of cement production. This may be secured by taking the entire test sample at a single operation, known as the "grab method," or by combining several portions taken at regular intervals, known as the "composite method." When obtaining a composite sample, it shall be secured by combining approximately equal weights of the cement taken at regular intervals, each portion representing approximately 40 bags. Automatic samplers may be used in obtaining samples.
>
> (*b*) *From Bulk Storage at Points of Discharge.*—Sufficient cement shall be drawn from the discharge openings to obtain samples representative of the cement as determined by the appearance at the discharge openings of indicators placed on the surface of the cement directly above these openings before the drawing of the cement is started. One 4-lb. sample shall be taken for at least each 2000 bags, by either the "grab" or "composite" method as described in Paragraph (*a*).
>
> (*c*) *From Bulk Storage by Means of Proper Sampling Tubes.*—When the methods described in Paragraphs (*a*) and (*b*) cannot be applied and when the depth of the cement to be sampled does not exceed 10 ft., samples may be obtained by proper tubes inserted vertically to the full depth of the cement. Samples so taken shall be obtained from points well distributed over the area of storage.
>
> (*d*) In all other cases, samples shall be taken from each 50 bags or portion thereof in the lot and combined to form test samples. In the case of samples from trucks where the cement is being trucked from one mill, it is permissible to combine the samples from several trucks to form a test sample representing not more than 2000 bags. When bulk shipments are sampled, representative samples shall be taken from well-distributed points.

III. ADOPTION OF TENTATIVE AS STANDARD

Tentative Methods of Chemical Analysis of Portland Cement (**C 114 – 44 T**).[4]—Sections 2 to 10 of these chemical methods have now stood for a considerable time without serious criticism, and the committee recommends that those sections be adopted as standard, and included in the Standard Methods of Chemical Analysis of Portland Cement (C 114 – 44). Those sections cover the following procedures: Rapid methods for sodium oxide and potassium oxide; method for sulfide sulfur; alternate method of rapid determination of sulfur trioxide by turbidimetry. It is recommended that Sections 11 to 13 (method for determining Vinsol resin in cement) be continued as tentative for another year.

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[4] 1944 Book of A.S.T.M. Standards, Part II.

TABLE II.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. REVISION OF TENTATIVE			
Spec. for Air-Entraining Portland Cement (C 175 – 46 T)	62½	2	6½
II. REVISION OF STANDARDS, IMMEDIATE ADOPTION			
Spec. for Portland Cement (C 150 – 44):			
Revision of Table I	62½	2	7½
Revision of Sec. 9(b)	66⅓	1	1⅓
Sampling Hydraulic Cement (C 183 – 44)	64⅓	0	4⅓
III. ADOPTION OF TENTATIVE AS STANDARD			
Chemical Analysis of Portland Cement (C 114 – 44 T), Sections 2 to 10 only:			
Rapid Alkali Method	59½	0	12½
Sulfide Sulfur	62½	0	9½
Turbidimetric Method for SO_3	61½	0	10½

The recommendations appearing in this report have been submitted to letter ballot of the committee, which consists of 83 voting members, with the results shown in Table II.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, F. H. Jackson.
Vice-Chairman, W. H. Klein.
Secretary, G. E. Warren.
Six Members at Large of the Advisory Committee,
  H. D. Baylor R. E. Roscoe
  R. F. Blanks C. H. Scholer
  W. C. Hanna C. E. Wuerpel

This report has been submitted to letter ballot of the committee, which consists of 83 voting members; 69 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

F. H. JACKSON,
Chairman.

G. E. WARREN,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-1 presented to the Society through the Administrative Committee on Standards a proposed tentative revision of the Standard Method of Test for Compressive Strength of Hydraulic Cement Mortars (C 109 – 44). This tentative revision was accepted by the Standards Committee on September 9, 1946, and appears in the 1946 Book of A.S.T.M. Standards, Part II, p. 1701.

APPENDIX I

SCOPE OF COOPERATIVE TESTS OF CEMENT MORTARS SPONSORED BY THE WORKING COMMITTEE ON STRENGTH

Standardization of Flow Tables.—Because of the variations in design, mounting, and method of operation of the 10-in. flow table, a means of standardization was tried. Detailed instructions for procedure were furnished. A sample of pulverized limestone was distributed to the laboratories for use in preparing the mortar to be used in the standardization. The mortar consisted of 525 g. of pulverized limestone, 1350 g. of graded Ottawa sand, and 230 ml. of mixing water.

Mixing of Mortars.—Variations in the method of preparing the mortars used in testing the cements included hand mixing, mechanical mixing, and "rubber ball" mixing.

Molding.—The molding methods included the procedure described in the Standard Method of Test for Compressive Strength of Hydraulic-Cement Mortars (C 109 – 44),[1] together with studies of effects of variations in tamping, and variations in the mixing, flow, and molding schedules.

Air Content of Mortars.—Three rounds of tests, one each on a different day, were made under each of four conditions:

(*1*) Tentative Method of Test for Air Content of Portland-Cement Mortar (C 185 – 44 T),[1] using the Burmister flow trough.

(*2*) Method C 185, but using the amount of water that would produce a flow between 90 and 110 per cent on the 10-in. flow table, when the table is dropped from a height of $\frac{1}{2}$ in., 10 times in about 7 sec.

(*3*) Method C 185, except using rubber ball mixer.

(*4*) Method C 185, except preparing mortar with a mechanical mixer of revolving blade type.

Miscellaneous.—Premature stiffening tests on cement; check of grading of the Ottawa sand; study of cube molds; data on the compression testing machines, mixing bowls, and flow tables.

Cements.—Six cements[2] were used, as follows: Type I, type IA with air content of about 12 per cent in standard mortar, type IA with about 16 per cent in the standard mortar, type II, type IIA, and type III.

Laboratories.—The following six laboratories participated in the tests:

Central Concrete Laboratory, U. S. Army, Corps of Engineers,
National Bureau of Standards,
Portland Cement Association,
Public Roads Administration,
U. S. Bureau of Reclamation, and
Universal Atlas Cement Co.

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[1] 1944 Book of A.S.T.M. Standards, Part II.

[2] Requirements for types I, II, and III are given in the Standard Specifications for Portland Cement (C 150 – 44), and for types IA and IIA in the Tentative Specifications for Air-Entraining Portland Cement (C 175 – 44 T), 1944 Book of A.S.T.M. Standards, Part II.

APPENDIX II

REPORT OF WORKING COMMITTEE ON SULFATE RESISTANCE

Based on the 1945 Lean Mortar Bar Tests

This report covers the 1945 lean mortar bar tests which were made by the following cooperators:

- California Highway Department, Sacramento, Calif.,
- Central Concrete Laboratory, U. S. Army, Corps of Engineers, Mt. Vernon, N. Y.,
- Cooperative Laboratory, University Farm, St. Paul, Minn.,
- Pennsylvania-Dixie Cement Corp., Nazareth, Pa.,
- Portland Cement Association, Chicago, Ill., and
- Public Roads Administration, Washington, D. C.

Note.—The laboratories are not listed in the same order in which they appear in the tables.

This work is a continuation of the lean mortar bar tests reported in 1943 and 1944 and published in 1945,[1] which tests were based on work of the following organizations:

- California Highway Department, Sacramento, Calif.,
- California Portland Cement Co., Colton, Calif.,
- Cooperative Laboratory, University Farm, St. Paul, Minn.,
- National Bureau of Standards, Washington, D. C., and
- Portland Cement Association, Chicago, Ill.

Chemical analyses and other essential data of the 1945 tests are shown in Tables I and II. Results of the lean mortar bar tests from all laboratories have been grouped in Tables III, IV, and V.

The laboratory temperatures and relative humidities at the time of fabrication of the lean mortar bars were reported by each of the six cooperating laboratories as follows:

Laboratory	Temperature, deg. Fahr.	Relative Humidity, per cent
A........	72 to 77	30 to 59
B..........	68.5 to 71.5	98 to 100
C...........	69 to 72.5	66 to 83
D...........	73 to 77	50
E..........	.....	45 to 50
F..........	70 to 76	36 to 66

The following discussion refers particularly to expansions of the bars at 42 days although it is closely applicable to expansions at either 28 or 56 days.

It will be noted that the bars from laboratory C expanded least while the bars from laboratory B expanded most. With the possible exception of the bars from laboratory B, no satisfactory explanation can be offered for variations in expansions reported by the six laboratories. The weights of the bars from laboratory B averaged from 7 to 16 g. lighter than the average of the bars from all six laboratories. This is shown by reference to Tables III, IV, and V. On the other hand, the bars from laboratory F were heaviest but had just about average expansions. All of this may be reduced to the general observation that for the purpose of this test extreme care must be taken to eliminate lightweight bars from consideration. It so happens that the bars from laboratory B were made under generally accepted ideal laboratory conditions of temperature and humidity for, as reported, all specimens were fabricated in the moist room; temperature 70 ± 1½ F., humidity 98 to 100 per cent. The specimens were fabricated as well as cured in the moist room in an effort to avoid any erratic results which might be attributable to variations in humidity and temperature. Such being the case, the reason for the bars from laboratory B averaging lighter in weight just about resolves itself into lack of compaction, due perhaps to somewhat lighter thumbing than was used at the other laboratories, which in turn is reflected by greater expansions in the sulfate solutions.

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[1] Report on Comparative Short-Time Tests for Sulfate Resistance of 121 Commercial Cements. Prepared by Dalton G. Miller and Charles G. Snyder as a Report of the Working Committee on Sulfate Resistance, Appendix III, Report of Committee C-1, *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 165 (1945).

However, if the expansions of the bars from laboratory B are entirely eliminated from consideration, reference to the 42-day tests shows need for a more uniform routine for the lean mortar bar test because, as now applied, the test is relative rather than absolute. By this is meant that a given laboratory, with given cements, can do a pretty good job of rating the cements for sulfate resistance. The difficulty arises when an attempt is made to compare length changes obtained by different laboratories. Undue discrepancies between expansions of bars made at different laboratories were noted in a number of the tests made in 1943 and 1944, as previously reported,[1] and was the primary reason for conducting the 1945 cooperative tests.

One cause for discrepancies of bar expansions reported by different laboratories may be lack of temperature control for the solutions in which the bars are stored. Experiences in laboratory F indicate that in order to secure consistent readings temperatures must be maintained closely around 70 F. It is doubted that satisfactory repeat readings are possible if the temperature range exceeds about plus or minus 1½ F.

The 1945 lean mortar tests were all made in accordance with the routine outlined in the 1943 report,[1] except at laboratories D and F. At these two laboratories all bars were thumbed on top, as the materials were placed, and then thumbed on the reverse side after turning over the molds. It is believed that the practice of thumbing the bars on the two sides is a definite improvement in the original routine calling for thumbing on the one side. At laboratory F, the molds and materials were placed on a 1000-lb. platform scale with the beam loaded to 20 lb. so that the pressures applied could be constantly checked and controlled. It was the practice to apply pressures with the thumbs until the scale beam clicked sharply as it reached the upper limit of swing, indicative of tamping pressures judged to be about 25 lb. This method of gaging and controlling the tamping pressures worked out quite satisfactorily although, in spite of all these precautions, some runs of bars were out of line with respect to weights and expansions in the solutions.

Unfortunately, even after the 1945 tests, the Working Committee on Sulfate Resistance finds itself still unable to prescribe how all discrepancies in the bars may be avoided, and because of this the following four lines of approach suggest themselves.

1. Make available to any laboratory limited quantities of a laboratory prepared cement to be used as a standard when testing cements for sulfate resistance. That this thought is not wholly illusionary is borne out by the fact that cement No. 799 tested best of the 10 cements at all 6 laboratories. Reference to Table I shows that cement No. 799 was the only one of the 10 cements that fully met the requirements for type V

cement of the Standard Specifications for Portland Cement (C 150 - 44).[2] Now, if there could be obtained a small quantity of a cement of known sulfate-resisting properties to be used in bars exposed along with the cements under test, then the lean mortar bar test, even as the bars are now made, would have definite and usable possibilities. It might even be feasible to use a commercial cement against which to check, providing agreement could be reached as to the qualifications for such a cement.

2. Investigate the possibilities of mechanical compaction of the materials as they are placed in the molds. Offhand it would seem that it should not be too difficult to devise some means either by the use of mechanically operated tampers, by applying pressure over the top surface of the bars, or by vibrating the materials into place, perhaps in conjunction with tamping or pressure application. Together with any studies of mechanical compaction of the materials in the molds, thought should also be given to improved standardization of mixing practices because, conceivably, some of the trouble now experienced with the lean mortar bars may be a reflection of improper mixing, somewhere along the line.

3. Explore possibilities of improving the uniformity of the bars by using somewhat wetter mixes. It is doubted that this, by itself, will eliminate differences between laboratories but merits consideration.

4. Make provisions for an operator, experienced in the making of lean mortar bars, to visit those laboratories in the United States that nominally have need for an acceptance test for sulfate resistance so that various operators could be instructed in the art of making uniform bars and in the various other details of the lean mortar bar test. It is strongly felt that a competent man could do much to bring about far greater uniformity in the results of the lean mortar test, even by the routine now generally followed.

Respectfully submitted on behalf of the Working Committee,

DALTON G. MILLER,
Chairman.

NOTE.—Since the preparation of the foregoing report, the University Farm laboratory has investigated the reproducibility of results when bars were molded under a pressure of 2000 lb. (320 psi.) applied and held for 10 sec., first to the upper surface of the mortar, and then, after reversal of the mold, to the opposite surface. Using this method of compaction, companion specimens were molded and comparisons made at each age with all of the specimens listed in Tables III and IV, except for cement No. 793.

The pressure method of molding was considered as much easier for the operator than was the thumbing of the bars, and was considered worthy of further study as a means of improving the reproducibility of procedure among different laboratories.

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[2] 1944 Book of A.S.T.M. Standards, Part II.

TABLE I.—CHEMICAL ANALYSES, CALCULATED COMPOSITION, AND MISCELLANEOUS TESTS OF 10 CEMENT STESTED FOR SULFATE RESISTANCE.

Data from Laboratory D.

Cement Number	Chemical Analyses, per cent												Loss on Ignition, per cent	Insoluble Residue, per cent
	SiO_2[a]	Al_2O_3[b]	Fe_2O_3[c]	CaO[d]	MgO[e]	SO_3	Na_2O	K_2O	FeO	TiO_2	P_2O_5	Mn_2O_3		
789	21.44	4.13	2.69	62.82	2.02	1.86	0.17	0.50	T.	0.24	0.15	0.69	1.72	0.19
790	21.91	4.47	3.24	64.95	1.54	1.72	0.18	0.36	T.	0.24	0.13	0.14	0.82	0.16
791	22.12	4.34	2.99	63.29	2.96	1.75	0.19	0.55	0.04	0.23	0.14	0.13	0.92	0.13
792	20.36	5.07	4.50	61.88	2.68	1.41	0.15	0.17	0.15	0.23	0.03	0.40	1.82	0.12
793	21.11	3.88	3.34	65.87	1.82	2.26	0.15	0.26	T.	0.22	0.10	0.10	0.91	0.12
794	20.99	5.60	3.24	62.35	3.06	1.72	0.35	0.55	T.	0.31	0.15	0.30	0.90	0.18
795	21.95	5.18	2.11	63.94	1.11	1.74	0.05	0.15	0.04	0.31	0.13	0.09	1.97	0.23
796	21.07	5.77	2.37	61.89	2.64	1.93	0.17	0.88	0.03	0.27	0.04	0.05	1.57	0.13
797	19.96	6.71	3.26	62.13	2.24	1.72	0.16	0.27	T.	0.28	0.03	0.95	1.33	0.16
799	24.79	3.35	3.75	63.05	1.40	1.49	0.34	0.35	0.03	0.16	0.09	0.10	0.77	0.08

Cement Number	Calculated Composition from Corrected Oxides, per cent					Free CaO, per cent	Chloroform-Soluble Matter, per cent	Passing No. 325 Sieve, per cent	Specific Surface (Wagner), sq. cm. per g.
	C_3S	C_2S	C_3A	C_4AF	$CaSO_4$				
789	55.9	19.4	6.4	8.2	3.2	0.29	0.004	96.1	1860
790	58.3	18.9	6.4	9.9	2.9	0.07	0.002	91.0	1740
791	51.1	24.9	6.4	9.1	3.0	0.07	0.003	91.4	1640
792	52.6	18.7	5.8	13.7	2.4	0.93	0.003	90.8	1990
793	70.4	7.4	4.6	10.2	3.8	0.04	0.002	99.5	2630
794	47.1	24.7	9.4	9.9	2.9	0.24	0.003	88.1	1750
795	50.7	24.7	10.2	6.4	3.0	1.14	0.003	85.8	1760
796	44.1	27.2	11.3	7.2	3.3	1.28	0.002	92.7	1630
797	46.5	22.1	12.3	9.9	2.9	0.87	0.004	91.1	1870
799	36.1	43.9	2.5	11.4	2.5	0.11	0.004	97.1	1860

[a] Corrected for insoluble residue.
[b] Corrected for TiO_2 and P_2O_5.
[c] Corrected for FeO as Fe_2O_3.
[d] Corrected for Free CaO.
[e] Corrected for Mn_2O_3
T Indicates a trace.

TABLE II.—AMOUNT OF MIXING WATER USED IN FABRICATION OF 1:5 OTTAWA STANDARD SAND BARS MADE OF 10 CEMENTS.

NOTE.—The results reported are of 6 bars, 5 in. in net length, made at 6 cooperating laboratories.

Cement Number	Normal Consistency						Mixing Water, ml.					
	Laboratory						Laboratory					
	A	B	C	D[a]	E	F	A	B	C	D[a]	E	F
789	24.5	24.0	24.5	24.5	27.0	24.5	133	132	133	263	137	133
790	24.0	23.6	24.5	24.5	25.0	24.5	132	131	133	263	134	133
791	23.5	23.0	23.0	23.0	24.5	23.0	131	130	130	258	133	130
792	23.0	23.6	23.5	23.5	23.5	23.5	130	131	131	260	131	131
793	27.0	26.0	26.5	25.25	27.0	26.0	137	135	136	265	137	135
794	23.5	24.0	24.0	24.0	24.0	24.0	131	132	132	261	132	132
795	23.5	23.6	24.0	24.0	24.5	24.0	131	131	132	261	133	132
796	25.0	26.0	26.0	25.5	26.0	26.0	134	135	135	266	135	135
797	23.5	24.0	24.5	24.5	24.5	24.5	131	132	133	263	133	133
799	22.0	23.0	23.0	23.0	23.0	23.0	128	130	130	258	130	130

[a] Laboratory D used a double batch of mortar and made 12 bars from each batch.

TABLE III.—INCREASES IN EXPANSION AND WEIGHT OF 1:5 OTTAWA STANDARD SAND BARS MADE OF 10 CEMENTS, BARS STORED IN WATER.

Note.—Bars 1 by 1 in. with 5-in. net length, stored for 56 days as indicated after curing 2 days in moist room and days in water. Data from 6 cooperating laboratories.

Laboratory	No. of Runs	No. of Bars	Bars Stored in Water and Measured at 7-day Intervals								Original Weight, g.	Increase in Weight, per cent	
			Expansion, per cent										
			7 days	14 days	21 days	28 days	35 days	42 days	49 days	56 days	2 days	28 days	56 days
							CEMENT 789						
A	3	6	−0.006	0.001	0.001	0.003	0.000	0.003	0.005	0.003	224	0.42	0.66
B	3	6	0.002	0.003	0.006	0.006	0.006	0.006	0.006	0.006	213	0.60	1.50
C	3	5	0.011	0.013	0.013	0.014	0.016	0.017	0.018	0.019	220	1.00	1.32
D	2	2	0.007	0.008	0.009	0.010	0.010	0.010	0.010	0.010	219	1.13	1.58
E	3	6	−0.016	−0.004	0.000	0.006	0.002	−0.002	0.000	0.000	220	0.70	1.60
F	4	6	0.003	0.007	0.009	0.008	0.009	0.008	0.008	0.010	229	1.67	2.14
Average			0.000	0.004	0.006	0.008	0.007	0.007	0.008	0.008	221	0.92	1.38
							CEMENT 790						
A	3	6	−0.009	0.000	0.000	−0.001	0.000	0.002	0.006	0.002	222	0.32	0.55
B	3	6	0.003	0.003	0.004	0.005	0.005	0.005	0.005	0.004	212	0.80	1.50
C	3	6	0.010	0.012	0.011	0.012	0.013	0.014	0.015	0.016	220	0.73	0.95
D	2	2	0.001	0.003	0.005	0.005	0.005	0.005	0.005	0.005	223	1.13	1.35
E	3	6	−0.002	0.000	0.000	0.006	0.004	0.004	0.004	0.000	218	0.50	1.50
F	4	6	0.001	0.008	0.007	0.008	0.008	0.008	0.006	0.011	230	1.16	1.48
Average			0.001	0.004	0.005	0.006	0.006	0.006	0.007	0.006	221	0.77	1.22
							CEMENT 791						
A	3	6	−0.002	−0.003	−0.001	−0.001	0.001	0.002	0.003	0.001	221	0.35	0.61
B	3	6	0.001	0.003	0.004	0.004	0.005	0.005	0.005	0.005	208	0.50	1.20
C	3	6	0.005	0.006	0.008	0.009	0.009	0.011	0.012	0.013	219	0.78	1.10
D	2	2	0.005	0.007	0.008	0.010	0.010	0.010	0.010	0.010	217	1.35	1.80
E	3	6	−0.004	0.004	0.004	0.006	0.004	0.008	0.006	0.000	220	0.40	1.30
F	4	6	0.005	0.008	0.010	0.012	0.012	0.011	0.012	0.013	228	1.28	1.54
Average			0.002	0.004	0.006	0.007	0.007	0.008	0.008	0.007	219	0.78	1.26
							CEMENT 792						
A	3	6	−0.001	0.005	0.005	0.002	0.007	0.009	0.006	0.007	222	0.47	0.64
B	3	6	0.004	0.004	0.005	0.006	0.007	0.007	0.006	0.006	210	0.80	1.70
C	3	6	0.005	0.006	0.009	0.010	0.010	0.011	0.012	0.013	221	0.45	0.63
D	2	2	0.002	0.004	0.004	0.004	0.004	0.006	0.006	0.006	219	1.13	1.58
E	3	6	−0.002	0.002	0.006	0.012	0.010	0.012	0.008	0.006	222	0.40	1.20
F	4	6	0.005	0.009	0.011	0.010	0.010	0.011	0.011	0.014	228	1.97	2.16
Average			0.002	0.005	0.007	0.007	0.008	0.009	0.008	0.009	220	0.87	1.32
							CEMENT 793						
A	3	6	0.001	0.002	0.002	0.002	0.007	0.012	0.008	0.006	225	0.29	0.44
B	3	6	0.001	0.001	0.002	0.002	0.002	0.002	0.000	0.000	211	0.50	1.50
C	3	6	0.004	0.006	0.007	0.007	0.008	0.009	0.010	0.012	223	0.58	0.85
D	2	2	0.006	0.006	0.006	0.006	0.006	0.006	0.006	0.007	224	1.13	1.35
E	3	6	−0.002	−0.002	0.002	0.006	0.004	0.006	0.000	−0.002	222	0.60	1.10
F	4	6	0.003	0.009	0.009	0.010	0.012	0.010	0.006	0.010	231	1.37	1.51
Average			0.002	0.004	0.005	0.007	0.007	0.008	0.005	0.006	223	0.74	1.13
							CEMENT 794						
A	3	6	−0.003	−0.001	0.001	0.002	0.006	0.010	0.009	0.008	223	0.60	0.94
B	3	6	0.002	0.006	0.006	0.007	0.007	0.008	0.008	0.008	211	1.00	2.20
C	3	6	0.005	0.008	0.009	0.009	0.011	0.013	0.014	0.016	220	0.82	1.23
D	2	2	0.004	0.004	0.006	0.006	0.007	0.007	0.009	0.010	218	1.58	2.03
E	3	6	−0.002	0.004	0.004	0.008	0.008	0.006	0.006	0.004	219	1.20	1.80
F	4	6	0.002	0.009	0.008	0.008	0.010	0.010	0.007	0.012	227	2.09	2.24
Average			0.001	0.005	0.006	0.007	0.008	0.009	0.009	0.010	220	1.22	1.74

TABLE III—*Concluded.*

Laboratory	No. of Runs	No. of Bars	Bars Stored in Water and Measured at 7-day Intervals								Original Weight, g.	Increase in Weight, per cent	
			Expansion, per cent										
			7 days	14 days	21 days	28 days	35 days	42 days	49 days	56 days	2 days	28 days	56 days
CEMENT 795													
A	3	6	0.002	0.001	0.006	0.006	0.007	0.009	0.007	0.010	220	0.53	0.77
B	3	6	0.002	0.003	0.004	0.007	0.006	0.007	0.006	0.005	211	0.90	1.80
C	3	6	0.006	0.008	0.009	0.010	0.011	0.012	0.014	0.016	220	0.77	1.18
D	2	2	0.004	0.004	0.004	0.004	0.006	0.006	0.006	0.006	222	1.13	1.35
E	3	6	−0.004	0.008	0.004	0.010	0.012	0.008	0.008	0.008	220	0.70	1.20
F	4	6	0.006	0.009	0.010	0.010	0.010	0.009	0.005	0.011	227	1.21	1.32
Average			0.003	0.006	0.006	0.008	0.009	0.009	0.008	0.009	220	0.87	1.27
CEMENT 796													
A	3	6	−0.001	0.000	0.000	0.005	0.007	0.008	0.007	0.008	222	0.62	0.97
B	3	6	0.002	0.004	0.004	0.006	0.006	0.006	0.005	0.005	212	0.90	1.70
C	3	6	0.006	0.007	0.009	0.010	0.013	0.014	0.017	0.020	219	0.78	1.14
D	2	2	0.002	0.002	0.002	0.002	0.002	0.003	0.003	0.003	217	1.35	1.80
E	3	6	−0.004	0.010	0.004	0.008	0.008	0.004	0.008	0.006	217	0.90	1.60
F	4	6	0.004	0.008	0.006	0.007	0.008	0.009	0.008	0.010	228	1.32	1.57
Average			0.002	0.005	0.004	0.006	0.007	0.007	0.008	0.009	219	0.98	1.46
CEMENT 797													
A	3	6	−0.002	−0.003	0.000	0.005	0.006	0.005	0.009	0.008	221	0.63	0.89
B	3	6	0.003	0.005	0.006	0.007	0.007	0.007	0.007	0.007	210	1.00	2.10
C	3	6	0.005	0.008	0.008	0.010	0.013	0.015	0.017	0.022	221	0.50	0.72
D	2	2	0.002	0.003	0.003	0.004	0.004	0.006	0.007	0.007	219	1.35	1.35
E	3	5	0.002	0.018	0.010	0.018	0.010	0.012	0.010	0.014	220	1.00	1.60
F	4	6	0.005	0.008	0.008	0.011	0.010	0.009	0.010	0.014	228	2.44	2.66
Average			0.003	0.007	0.006	0.009	0.008	0.009	0.010	0.012	220	1.15	1.55
CEMENT 799													
A	3	6	0.001	0.004	0.003	0.005	0.007	0.004	0.009	0.006	223	0.16	0.56
B	3	6	0.003	0.005	0.006	0.006	0.006	0.008	0.007	0.007	212	0.80	1.60
C	3	6	0.006	0.007	0.009	0.011	0.013	0.015	0.017	0.022	221	0.54	0.91
D	2	2	0.000	0.000	0.000	0.001	0.002	0.002	0.002	0.002	220	1.35	1.80
E	3	6	0.000	0.016	0.008	0.014	0.012	0.008	0.008	0.004	220	0.50	1.20
F	4	6	0.005	0.009	0.012	0.012	0.011	0.012	0.011	0.013	228	0.95	1.13
Average			0.003	0.007	0.006	0.008	0.009	0.008	0.009	0.009	221	1.06	1.20

TABLE IV.—INCREASES IN EXPANSION AND WEIGHT OF 1:5 OTTAWA STANDARD SAND BARS MADE OF 10 CEMENTS, BARS STORED IN SODIUM SULFATE SOLUTIONS.

NOTE.—Bars 1 by 1 in. with 5-in. net length, stored for 56 days as indicated after curing 2 days in moist room and days in water. Data from 6 cooperating laboratories.

Laboratory	No. of Runs	No. of Bars	Bars Stored in 0.15*M* (2.1 per cent) Sodium Sulfate Solutions and Measured at 7-day Intervals								Original Weight, g.	Increase in Weight, per cent	
			Expansion, per cent										
			7 days	14 days	21 days	28 days	35 days	42 days	49 days	56 days	2 days	28 days	56 days
CEMENT 789													
A	3	6	0.003	0.004	0.012	0.011	0.012	0.015	0.019	0.023	224	0.39	0.44
B	3	6	0.006	0.009	0.013	0.017	0.024	0.030	0.037	0.048	209	1.00	1.60
C	3	6	0.013	0.015	0.018	0.020	0.024	0.026	0.028	0.031	221	0.63	0.72
D	5	5	0.008	0.010	0.014	0.014	0.014	0.016	0.018	0.020	220	0.45	0.45
E	3	6	−0.002	0.002	0.010	0.016	0.018	0.024	0.022	0.022	221	1.00	1.70
F	4	6	0.006	0.010	0.010	0.017	0.019	0.020	0.019	0.024	229	1.71	1.97
Average			0.006	0.008	0.013	0.016	0.019	0.022	0.024	0.028	221	0.86	1.15
CEMENT 790													
A	3	6	−0.002	0.006	0.009	0.011	0.013	0.019	0.021	0.028	222	0.22	0.35
B	3	6	0.008	0.010	0.014	0.021	0.030	0.045	0.066	0.101	206	0.90	2.00
C	3	6	0.011	0.013	0.015	0.017	0.020	0.022	0.025	0.028	219	0.64	0.73
D	5	5	0.000	0.004	0.004	0.004	0.005	0.005	0.008	0.010	224	0.45	0.54
E	3	6	−0.002	0.006	0.012	0.018	0.038	0.062	0.088	0.132	218	1.00	1.60
F	4	6	0.005	0.012	0.013	0.014	0.018	0.018	0.018	0.025	229	1.13	1.60
Average			0.003	0.009	0.011	0.014	0.021	0.029	0.038	0.054	220	0.72	1.14
CEMENT 791													
A	3	6	−0.005	0.005	0.005	0.005	0.006	0.010	0.014	0.017	222	0.23	0.41
B	3	6	0.007	0.012	0.018	0.031	0.051	0.075	0.104	0.150	205	1.10	2.10
C	3	6	0.004	0.007	0.011	0.012	0.014	0.015	0.018	0.019	218	0.41	1.01
D	5	5	0.007	0.010	0.011	0.013	0.013	0.014	0.016	0.019	216	0.36	0.45
E	3	6	0.006	0.006	0.014	0.018	0.016	0.024	0.022	0.026	218	0.70	1.40
F	4	6	0.008	0.012	0.014	0.017	0.019	0.021	0.018	0.024	229	1.28	1.75
Average			0.005	0.009	0.012	0.016	0.020	0.027	0.032	0.043	218	0.68	1.19
CEMENT 792													
A	3	6	0.007	0.014	0.018	0.022	0.041	0.074	0.094	0.129	222	0.49	0.77
B	3	6	0.009	0.016	0.029	0.055	0.093	0.143	0.207	0.288	206	1.00	2.60
C	3	6	0.005	0.009	0.013	0.016	0.017	0.018	0.022	0.024	222	0.32	0.50
D	5	5	0.004	0.008	0.009	0.009	0.013	0.018	0.021	0.031	220	0.54	0.63
E	3	6	0.004	0.012	0.066	0.140	0.226	0.332	0.438	0.518	220	1.10	2.50
F	4	6	0.007	0.016	0.021	0.025	0.031	0.039	0.060	0.101	228	2.16	2.64
Average			0.006	0.013	0.026	0.045	0.070	0.104	0.140	0.182	220	0.94	1.61
CEMENT 793													
A	3	6	0.003	0.005	0.010	0.016	0.024	0.033	0.047	0.070	226	0.17	0.24
B	3	6	0.008	0.013	0.028	0.054	0.092	0.141	0.202	0.293	209	1.20	2.10
C	3	6	0.006	0.009	0.010	0.010	0.012	0.014	0.016	0.017	223	0.31	0.54
D	5	5	0.008	0.008	0.008	0.008	0.008	0.015	0.020	0.024	225	0.35	0.53
E	3	6	0.000	0.008	0.014	0.034	0.050	0.118	0.206	0.298	224	0.80	1.50
F	4	6	0.004	0.012	0.014	0.014	0.016	0.019	0.017	0.032	233	1.25	1.47
Average			0.005	0.009	0.014	0.023	0.034	0.057	0.085	0.122	223	0.68	1.06
CEMENT 794													
A	3	6	0.008	0.014	0.023	0.045	0.077	0.133	0.208	0.302	223	0.33	0.65
B	3	6	0.008	0.015	0.030	0.055	0.104	0.177	0.279	0.405	206	1.80	2.90
C	3	6	0.007	0.013	0.016	0.017	0.020	0.024	0.027	0.031	220	0.54	0.77
D	5	5	0.006	0.008	0.012	0.017	0.029	0.052	0.086	0.120	219	0.27	0.63
E	3	6	0.000	0.004	0.028	0.094	0.204	0.372	0.572	0.788	220	1.20	2.50
F	4	6	0.007	0.019	0.023	0.025	0.041	0.078	0.141	0.238	228	2.02	2.64
Average			0.006	0.012	0.022	0.042	0.079	0.139	0.219	0.314	219	1.03	1.72

TABLE IV—*Concluded.*

Laboratory	No. of Runs	No. of Bars	Bars stored in 0.15*M* (2.1 per cent) Sodium Sulfate Solutions and Measured at 7-day Intervals								Original Weight, g.	Increase in Weight, per cent	
			Expansion, per cent										
			7 days	14 days	21 days	28 days	35 days	42 days	49 days	56 days	2 days	28 days	56 days
CEMENT 795													
A	3	6	0.009	0.010	0.020	0.035	0.055	0.089	0.126	0.174	221	0.37	0.70
B	3	6	0.007	0.016	0.025	0.039	0.059	0.089	0.137	0.193	206	1.30	2.40
C	3	6	0.008	0.012	0.015	0.016	0.020	0.024	0.027	0.030	220	0.50	0.82
D	5	5	0.004	0.006	0.008	0.011	0.015	0.020	0.026	0.034	226	0.44	0.53
E	3	6	−0.004	0.008	0.024	0.048	0.082	0.130	0.190	0.264	220	1.10	1.70
F	4	6	0.011	0.016	0.019	0.024	0.030	0.032	0.043	0.050	229	1.21	1.61
Average			0.006	0.011	0.019	0.029	0.044	0.064	0.092	0.124	220	0.82	1.29
CEMENT 796													
A	3	6	0.002	0.008	0.013	0.035	0.080	0.187	R	. . .	222	0.50	R
B	3	6	0.007	0.014	0.027	0.055	0.103	0.203	0.408	R	206	1.40	R
C	3	6	0.008	0.013	0.017	0.022	0.030	0.041	0.061	0.094	219	1.05	1.69
D	5	5	0.004	0.005	0.010	0.012	0.022	0.046	0.075	0.141	219	0.73	1.45
E	3	6	0.000	0.008	0.016	0.052	0.122	0.264	0.516	0.846+	220	1.00	2.40+
F	4	6	0.011	0.019	0.021	0.033	0.068	0.169	0.356	0.716	229	1.71	2.40
Average			0.005	0.011	0.017	0.035	0.072	0.152	. . .	. . . .	219	1.07	. . . .
CEMENT 797													
A	3	6	0.003	0.018	0.090	0.298	R	. . .	. . .	. . .	222	0.68	R
B	3	6	0.015	0.053	0.166	0.421	0.881	R	. . .	. . .	207	2.20	R
C	3	6	0.008	0.017	0.031	0.052	0.095	0.169	R	. . .	221	0.59	1.90+
D	5	5	0.005	0.011	0.026	0.061	0.156	0.371	0.747	R	219	0.90	R
E	3	6	0.004	0.028	0.184	0.708	1.468+	2.332+	3.228+	R	222	1.40	R
F	4	6	0.013	0.029	0.084	0.315	0.984+	2.371+	. . . .	. . . .	229	3.68	8.35+
Average			0.008	0.026	0.097	0.309	. . . .	. . . .	. . . .	. . . .	220	1.58	. . . .
CEMENT 799													
A	3	6	0.001	0.005	0.011	0.014	0.015	0.016	0.018	0.020	223	0.20	0.30
B	3	6	0.006	0.010	0.011	0.015	0.017	0.017	0.020	0.024	208	0.60	1.20
C	3	6	0.007	0.010	0.012	0.015	0.017	0.020	0.023	0.025	221	0.68	0.81
D	5	5	0.002	0.002	0.004	0.005	0.006	0.008	0.010	0.012	219	0.45	0.54
E	3	6	−0.002	0.000	0.004	0.008	0.018	0.016	0.020	0.022	221	0.60	1.20
F	4	6	0.014	0.015	0.016	0.016	0.018	0.018	0.017	0.026	229	0.88	1.02
Average			0.00[illegible]	0.007	0.010	0.012	0.015	0.016	0.018	0.022	220	0.57	0.85

R indicates failure.

TABLE V.—INCREASES IN EXPANSION AND WEIGHT OF 1:5 OTTAWA STANDARD SAND BARS MADE OF 10 CEMENTS, BARS STORED IN MAGNESIUM SULFATE SOLUTIONS.

NOTE.—Bars 1 by 1 in. with 5-in. net length, stored for 56 days as indicated after curing 2 days in moist room and days in water. Data from 6 cooperating laboratories.

Laboratory	No. of Runs	No. of Bars	Bars Stored in 0.15M (1.8 per cent) Magnesium Sulfate Solutions and Measured at 7-day Intervals								Original Weight, g.	Increase in Weight, per cent	
			Expansion, per cent										
			7 days	14 days	21 days	28 days	35 days	42 days	49 days	56 days	2 days	28 days	56 days
						CEMENT 789							
A	3	6	−0.001	0.006	0.012	0.014	0.013	0.019	0.022	0.024	223	1.05	1.36
B	6	6	0.007	0.013	0.020	0.036	0.058	0.085	0.112	0.140	207	1.80	3.30
C	3	6	0.011	0.015	0.017	0.019	0.021	0.024	0.026	0.029	220	0.95	1.13
D	5	5	0.001	0.012	0.013	0.016	0.021	0.022	0.024	0.026	223	0.90	0.99
E	3	6	−0.004	0.010	0.016	0.012	0.026	0.030	0.030	0.036	220	1.00	1.50
F	4	6	0.005	0.016	0.019	0.019	0.020	0.020	0.021	0.028	230	1.74	1.99
Average			0.003	0.012	0.016	0.019	0.027	0.033	0.039	0.047	222	1.24	1.70
						CEMENT 790							
A	3	6	0.003	0.007	0.009	0.013	0.012	0.018	0.025	0.032	222	0.91	1.22
B	3	6	0.005	0.011	0.020	0.053	0.080	0.122	0.167	0.215	205	2.20	3.30
C	3	6	0.008	0.010	0.011	0.014	0.016	0.016	0.018	0.022	220	0.59	0.91
D	5	5	0.000	0.010	0.011	0.014	0.018	0.020	0.024	0.027	222	0.99	0.99
E	3	6	−0.004	0.008	0.020	0.030	0.042	0.068	0.086	0.116	222	0.90	1.60
F	4	6	0.002	0.015	0.016	0.016	0.018	0.019	0.019	0.032	230	1.56	1.89
Average			0.002	0.010	0.015	0.023	0.031	0.042	0.057	0.074	220	1.19	1.65
						CEMENT 791							
A	3	6	0.002	0.009	0.012	0.012	0.019	0.019	0.024	0.025	221	0.91	1.21
B	3	6	0.006	0.013	0.019	0.033	0.055	0.085	0.122	0.163	206	2.10	3.00
C	3	6	0.003	0.006	0.009	0.010	0.011	0.013	0.017	0.018	220	0.73	1.37
D	5	5	0.001	0.010	0.012	0.015	0.021	0.022	0.025	0.028	221	1.08	0.99
E	3	6	−0.004	0.004	0.010	0.014	0.022	0.032	0.032	0.044	220	0.90	1.60
F	4	6	0.007	0.016	0.017	0.018	0.021	0.023	0.022	0.032	227	1.54	1.98
Average			0.002	0.010	0.013	0.017	0.025	0.032	0.040	0.052	219	1.21	1.69
						CEMENT 792							
A	3	6	0.002	0.012	0.014	0.017	0.023	0.038	0.046	0.057	222	1.03	1.31
B	3	6	0.012	0.035	0.094	0.168	0.247	0.328	0.384	0.446	204	3.10	4.50
C	3	6	0.005	0.008	0.012	0.014	0.015	0.017	0.021	0.022	222	0.36	0.95
D	5	5	0.009	0.012	0.013	0.018	0.024	0.027	0.032	0.038	222	1.08	1.17
E	3	6	0.008	0.020	0.068	0.128	0.198	0.258	0.320	0.370	220	1.40	1.80
F	4	6	0.011	0.019	0.024	0.034	0.060	0.094	0.128	0.198	228	2.67	3.14
Average			0.008	0.018	0.038	0.063	0.095	0.127	0.155	0.189	220	1.61	2.15
						CEMENT 793							
A	3	6	0.004	0.004	0.008	0.014	0.028	0.041	0.051	0.069	226	0.92	1.20
B	3	6	0.007	0.018	0.040	0.080	0.134	0.169	0.224	0.277	209	2.00	2.80
C	3	6	0.006	0.009	0.010	0.011	0.014	0.017	0.019	0.023	223	0.67	1.03
D	5	5	0.006	0.008	0.008	0.011	0.016	0.017	0.020	0.021	223	0.54	0.81
E	3	6	−0.004	0.008	0.012	0.036	0.084	0.140	0.198	0.262	222	0.80	1.30
F	4	6	0.011	0.016	0.018	0.018	0.025	0.032	0.035	0.059	233	1.64	1.82
Average			0.005	0.011	0.016	0.028	0.049	0.069	0.091	0.135	223	1.10	1.49
						CEMENT 794							
A	3	6	0.001	0.006	0.017	0.037	0.068	0.017	0.153	0.211	224	1.03	1.44
B	3	6	0.010	0.023	0.079	0.173	0.284	0.403	0.528	0.658	205	2.20	3.70
C	3	5	0.008	0.011	0.015	0.017	0.020	0.024	0.030	0.035	221	0.63	1.22
D	5	5	0.009	0.014	0.018	0.030	0.058	0.088	0.135	0.189	218	0.90	1.26
E	3	6	0.024	0.056	0.138	0.276	0.424	0.586	0.742	0.900	219	1.70	2.20
F	4	6	0.013	0.024	0.026	0.041	0.061	0.118	0.183	0.279	228	1.90	2.48
Average			0.011	0.022	0.049	0.096	0.152	0.221	0.295	0.379	219	1.39	2.05

TABLE V—*Concluded.*

Laboratory	No. of Runs	No. of Bars	Bars Stored in 0.15 *M* (1.8 per cent) Magnesium Sulfate Solutions and Measured at 7-day Intervals								Original Weight, g.	Increase in Weight, per cent	
			Expansion, per cent										
			7 days	14 days	21 days	28 days	35 days	42 days	49 days	56 days	2 days	28 days	56 days
			CEMENT 795										
A	3	6	0.000	0.005	0.021	0.037	0.062	0.099	0.133	0.175	219	1.24	1.67
B	3	6	0.007	0.017	0.040	0.094	0.164	0.249	0.337	0.430	205	2.10	3.60
C	3	5	0.008	0.012	0.015	0.016	0.020	0.023	0.027	0.032	220	0.73	1.14
D	5	5	0.006	0.008	0.010	0.013	0.016	0.016	0.020	0.022	220	0.45	0.90
E	3	6	0.010	0.018	0.040	0.084	0.134	0.194	0.256	0.316	220	1.10	1.70
F	4	6	0.009	0.017	0.020	0.023	0.025	0.044	0.056	0.089	227	1.51	1.94
Average			0.007	0.013	0.024	0.045	0.070	0.104	0.138	0.177	218	1.19	1.83
			CEMENT 796										
A	3	6	0.000	0.004	0.014	0.045	0.086	0.164	0.277	0.436	222	1.08	1.76
B	3	6	0.008	0.018	0.043	0.112	0.222	0.372	0.548	0.762	205	1.60	3.20
C	3	6	0.008	0.012	0.015	0.020	0.029	0.043	0.068	0.110	219	1.05	1.83
D	5	5	0.009	0.012	0.016	0.027	0.046	0.079	0.128	0.172	217	1.26	1.62
E	3	6	0.016	0.016	0.024	0.078	0.156	0.282	0.448	0.644	219	0.90	1.80
F	4	6	0.009	0.017	0.020	0.027	0.052	0.119	0.201	0.348	228	1.57	2.45
Average			0.008	0.013	0.022	0.052	0.099	0.177	0.278	0.412	218	1.24	2.11
			CEMENT 797										
A	3	6	0.000	0.013	0.049	0.122	0.246	0.427	0.677	1.007	222	1.09	2.17
B	3	6	0.016	0.069	0.193	0.367	0.561	0.777	0.997	R	206	2.50	R
C	3	6	0.008	0.014	0.019	0.030	0.049	0.072	0.113	0.182	223	0.63	1.34
D	5	5	0.010	0.016	0.032	0.075	0.180	0.391	0.766	1.291	220	0.54	2.81
E	3	6	0.012	0.078	0.246	0.596	0.960	1.356	1.796	2.220	222	1.60	3.20
F	4	6	0.017	0.029	0.110	0.322	0.729	1.278	1.712	2.196	229	3.20	5.53
Average			0.011	0.037	0.108	0.252	0.454	0.717	1.010	...	220	1.59	...
			CEMENT 799										
A	3	6	0.000	0.004	0.013	0.014	0.015	0.012	0.017	0.018	223	0.75	1.01
B	3	6	0.006	0.015	0.023	0.034	0.039	0.051	0.064	0.074	206	1.30	2.40
C	3	6	0.007	0.009	0.011	0.014	0.016	0.018	0.021	0.024	221	0.72	1.09
D	5	5	0.010	0.012	0.014	0.020	0.020	0.022	0.021	0.022	217	0.72	0.72
E	3	6	0.010	0.014	0.014	0.018	0.014	0.022	0.026	0.026	220	1.00	1.50
F	4	6	0.008	0.016	0.016	0.018	0.021	0.021	0.020	0.025	228	0.95	1.17
Average			0.005	0.012	0.015	0.020	0.021	0.024	0.028	0.032	219	0.91	1.32

R indicates failure.

APPENDIX III

PROPOSED METHOD OF TEST FOR SULFATE RESISTANCE OF PORTLAND CEMENT[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method of test is intended for determining the sulfate resistance of portland cement by length changes of lean mortar bars exposed to 0.15 *M* solutions of both sodium and magnesium sulfate.

Apparatus

2. (*a*) *Scales.*—The scales used in weighing materials for the mortar mixes shall conform to the following requirements: On scales in use the permissible variation at a load of 1000 g. shall be plus or minus 1.0 g., and at a load of 1500 g. shall be plus or minus 2.0 g. The permissible variations on new scales shall be one half of these values. The sensibility reciprocal[2] shall be not greater than twice the permissible variation.

(*b*) *Weights.*—The permissible variation on weights in use in weighing materials for the mortar mixes shall be as described in Table I. The permissible variations on new weights shall be one half of the values in Table I.

(*c*) *Sieves.*—Square-hole, woven wire cloth, No. 20 (840-micron) and No. 30 (590-micron) sieves conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11)[3] shall be used.

(*d*) *Glass Graduates.*—Glass graduates of suitable capacities (preferably large enough to measure the mixing water in a single operation) shall be made to deliver the indicated volume at 20 C. (68 F.). The permissible variation shall be plus or minus 1.0 ml. The graduates shall be subdivided to at least 1 ml. The main graduation lines shall be circles and shall be numbered. The least graduations shall extend at least one seventh of the way around, and intermediate graduations shall extend at least one fifth of the way around.

TABLE I.—PERMISSIBLE VARIATIONS ON WEIGHTS.

Weight, g.	Permissible Variations on Weights in Use, plus or minus, g.
1000	0.5
900	0.45
750	0.4
500	0.35
300	0.3
250	0.25
200	0.20
100	0.15
50	0.10
20	0.05
10	0.04
5	0.03
2	0.02
1	0.01

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee C-1 on Cement. Published as information, June, 1946.

[2] The sensibility reciprocal is a measure of the sensitiveness of a balance, and is the weight required to move the position of equilibrium of the pointer one division. For a complete definition of sensibility reciprocal, see "Specifications, Tolerances, and Regulations for Commercial Weighing and Measuring Devices," *Handbook H29*, Nat. Bureau Standards, September, 1942, pp. 87 and 88.

[3] 1946 Book of A.S.T.M. Standards, Part II.

(e) *Trowel.*—The trowel shall have a steel blade and be of convenient size.

(f) *Brush.*—A 1-in. flat brush with steel wire bristles.

(g) *Molds.*—Bar molds as shown in Fig. 1.

Temperature and Humidity

3. In view of the relatively long time required to prepare these bars, it is highly essential that, as closely as possible throughout the whole operation, the room temperature shall be maintained at 70 ± 1.5 F. and the relative humidity at not less than 50 per cent.

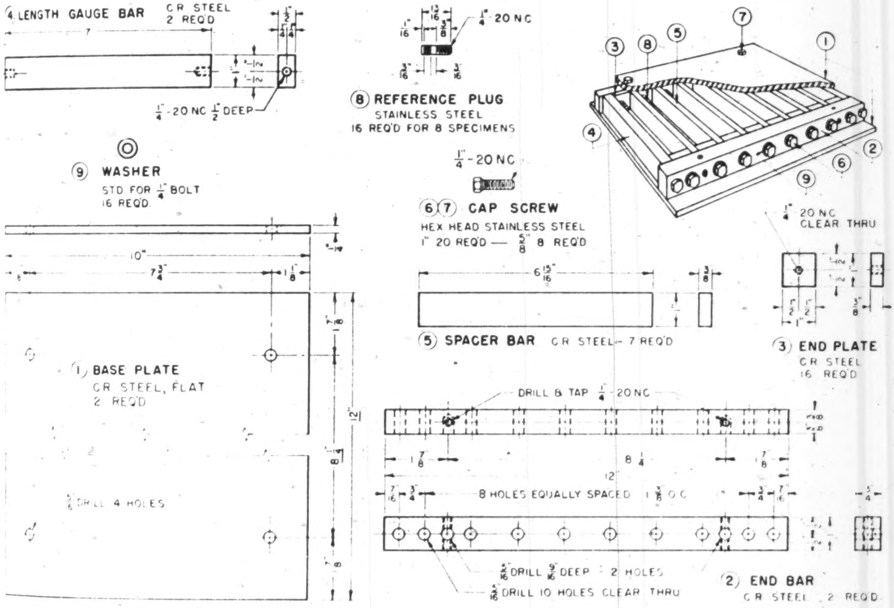
④ LENGTH GAUGE BAR
C.R. STEEL 2 REQ'D
7
1/4-20 NC 1/2 DEEP
⑨ WASHER
STD FOR 1/4 BOLT
16 REQ'D
10"
7 3/4"
1 1/8"
12"
① BASE PLATE
C.R. STEEL, FLAT
2 REQ'D
5/16 DRILL 4 HOLES
1/4-20 NC
⑧ REFERENCE PLUG
STAINLESS STEEL
16 REQ'D FOR 8 SPECIMENS
1/4-20 NC
⑥⑦ CAP SCREW
HEX HEAD STAINLESS STEEL
1" 20 REQ'D — 5/8" 8 REQ'D
6 15/16"
⑤ SPACER BAR C.R. STEEL — 7 REQ'D
1/4" 20 NC CLEAR THRU
③ END PLATE
C.R. STEEL
16 REQ'D
DRILL & TAP 1/4-20 NC
1 7/8
8 1/4
12"
8 HOLES EQUALLY SPACED 1 3/8 O.C.
5/16 DRILL 9/16 DEEP 2 HOLES
5/16 DRILL 10 HOLES CLEAR THRU
② END BAR
C.R. STEEL 2 REQ'D
①
②
③
④
⑤
⑥
⑦
⑧
⑨

FIG. 1.—Mold for Mortar Bar Specimens

Standard Sand

4. The sand used for making test specimens shall be natural silica sand from Ottawa, Ill., graded to pass a No. 20 (840-micron) sieve and retained on a No. 30 (590-micron) sieve. This sand shall be considered standard when not more than 15 g. are retained on a No. 20 sieve, and not more than 5 g. pass the No. 30 sieve after 5 min. of continuous sieving of a 100-g. sample in the manner specified for sieving cement in the Standard Method of Test for Fineness of Hydraulic Cement by the No. 200 Sieve (A.S.T.M. Designation: C 184).[3]

Test Specimens

5. The test specimens shall be bars 1 by 1 in. in cross-section, with a 5-in. effective gage length, and shall be made of cement and Ottawa standard sand (20–30). The test specimens shall be made in batches of six each.

Preparation of Mold

6. The molds shall be thinly coated with petroleum jelly or wax, assembled, and stainless steel reference plugs ($\frac{1}{4}$ by $\frac{13}{16}$ in.) shall be screwed into place in the removable 1 by 1-in. end plates, care being taken to keep the plugs clean and free of oil, grease or wax. A 1 by 1-in. piece of cardboard (Note 1) with a hole $\frac{1}{4}$ in. in diameter at the center shall then

be placed at each end of a mold. A layer of neat cement, of fairly stiff consistency, $\frac{1}{4}$ in. in thickness, shall be pressed against the oiled cardboard at each end of a mold (Note 2).

NOTE 1.—Any glazed cardboard about $\frac{1}{64}$ in. in thickness, soaked in oil and allowed to drain overnight, is satisfactory for this purpose.

NOTE 2.—A piece of $\frac{1}{8}$-in. strap iron with a U-shaped opening at one end is useful in placing and smoothing this neat cement cap. The thickness of the cap may be closely controlled by gaging with a piece of thin sheet metal held across the top of the mold at the ends while placing the neat cement.

Proportioning, Consistency, and Mixing of Mortars

7. (*a*) The proportions of the standard mortar shall be 1 part cement to 5 parts standard sand by weight. The percentage of water used in the standard mortar shall be as calculated from the normal consistency (Note 3) of the cement by the following equation:

$$y = \frac{2}{3}\,\frac{P}{n + 1} + K$$

where:

y = percentage of water required for the sand mortar,

P = percentage of water required for neat cement paste of normal consistency, determined in accordance with the Standard Method of Test for Normal Consistency of Hydraulic Cement (A.S.T.M. Designation: C 187),[3]

n = number of parts of sand to one of cement by weight, and

K = a constant which for the standard sand has the value 6.5.

Each batch of six bars shall consist of 1200 g. of sand, 240 g. of cement, and the calculated amount of water.

NOTE 3.—For a cement of 25 per cent normal consistency, the calculated quantity of mixing water is 134 ml. (9.3 per cent of the weight of the dry materials).

(*b*) The dry materials shall be weighed, placed upon a glass or other nonabsorbent flat surface, thoroughly mixed for $1\frac{1}{2}$ min. (Note 4), and a crater formed in the center. The proper quantity of distilled water, as calculated, shall be poured into the crater, and the material on the outer edge shall be turned into the crater within 30 sec. by the aid of a trowel. After an additional interval of 30 sec. for the absorption of the mixing water (Note 4) during which interval the dry mortar around the outside of the cone shall be lightly troweled over the remaining mortar to reduce the evaporation losses and to promote absorption, the operation shall be completed by continuous, vigorous mixing, squeezing, and kneading with the hands for $1\frac{1}{2}$ min. (Note 4). During the operation of mixing, the hands shall be protected by snug-fitting rubber gloves.

Molding Test Specimens

8. Immediately after completion of mixing, the mortar shall be placed in the molds (Note 4) by one of the following procedures:

(*a*) *Short-Time Molding.*—About $\frac{3}{4}$ in. of mortar shall be pressed into the neat cement, already in place, around the reference plug at each end of the mold. Each end of each mold shall receive 6 tamps of the index finger of the right hand, directed toward the end of the mold, while the index finger of the left hand holds the mortar from shoving out the top of the mold. The remaining mortar shall then be tamped into the mold in two layers, the long axes of the bars perpendicular to the front edge of the work table. The bottom layer shall be given 12 tamps with both thumbs working together, *ends* pointed directly downward, 6 tamps being distributed endwise along a bar in one direction, and the other 6 tamps being likewise distributed, but in the opposite direction. After rotating the mold 180 deg., the top layer shall then be tamped in a similar manner, except that, following the first 6 tamps, the excess material shall be redistributed with the trowel and then the last 6 tamps made with the *face* of the thumb. A uniform tamping pressure of 20 to 25 lb. shall be maintained as closely as possible through these operations. The excess materials shall be removed from the top and the mold with a straight edge and each bar finished with two strokes of the trowel exerting a pressure of approximately 5 lb. The mold shall then be covered with waxed paper, held in place by a sheet metal cover, and immediately placed in the moist closet.

(*b*) *Long-Time Molding.*—About $\frac{3}{4}$ in. of mortar shall be pressed into the neat cement, already in place, around the reference plug at each end of the mold. Each end of each mold shall receive 6 tamps of the index finger of the right hand directed toward the end of the mold while the index finger of the left hand holds the mortar from shoving out the top of the mold. The remaining mortar shall then be tamped into the molds in a single layer, the long axes of the bars perpendicular to the front edge of the work table Each bar shall then be tamped with both thumbs working together, *ends* pointed directly downward, 6 tamps being distributed endwise along a bar in one direction and 6 tamps being likewise distributed, but in the opposite direction. The mold shall then be rotated 180 deg., filled where necessary, and tamped 6 times with the *face* of the thumbs. The excess material shall then be removed, a top plate clamped on and the molds turned over. The bars shall then be tamped 6 times forward and 6 times backward with the *ends* of the thumbs, the mold rotated 180 deg., filled where necessary, tamped 6 times with the *face* of the thumbs and struck off (Note 5). The excess material shall be removed from the top of the mold with a straight edge and each bar finished with two strokes of a trowel exerting a pressure of approximately 5 lb. The mold shall be covered with waxed paper, held in place by a sheet metal cover, and immediately placed in the moist closet.

NOTE 4.—The entire routine of mixing and molding will take about 8 or 12 min. distributed over the various operations as follows:

Mixing dry materials	$1\frac{1}{2}$ min.
Absorption of mixing water	1 min.
Mixing wet materials	$1\frac{1}{2}$ min.
Placing in molds (Short-Time Method, Section 8 (*a*))	4 min.
Placing in molds (Long-Time Method, Section 8 (*b*))	8 min.

NOTE 5.—This is a total for each side of each bar of 12 tamps with the ends of the thumbs and 6 tamps with the face of the thumbs.

Storage of Test Specimens

9. All bars shall be held in molds in the moist closet for 46 to 48 hr. Immediately after removal from the molds, identification tags, or marks, shall be placed on individual bars and the bars weighed and measured and placed in fresh water for five days. All bars shall be weighed when taken from the moist room and any bar that is badly out of line in weight should be discarded. Individual bars should weigh not less than 220 g. and generally will weigh under 225 g. (Note 6).

NOTE 6.—Using slightly different water ratios, based on normal consistencies as determined, may occasionally cause bars of some of the cements to differ slightly from the average.

Procedure

10. (*a*) *Storage in Test Solutions.*—When the bars are seven days old (two days in moist room, five days in fresh water), they shall be measured and then placed in the test solutions. Solutions

shall be held in covered containers and laboratory temperatures maintained at 70 ± 1.5 F. Duplicate bars from each batch shall be stored in each of the following solutions and a test for sulfate resistance shall consist of no fewer than six bars made on three different days:

(*1*) Fresh water,

(*2*) Sodium sulfate, 0.15 *M* (2.1 per cent), and

(*3*) Magnesium sulfate, 0.15 *M* (1.8 per cent).

Solutions shall be renewed by changing completely every seven days. Bars shall be stored in the solutions in horizontal positions on slatted racks of wood or non-corrosive metal. Bars shall be so racked as to be approximately 1 in. from the bottom of a container with at least ¼ in. between bars. A ratio of solution to volume of bars in any container shall be maintained at about 4 to 1.

(*b*) *Measurement of Resistance to Test Solutions.*—The bars shall be measured at intervals of 7 days. They shall be weighed after exposure to the solutions for 28 days, when the bars are 35 days old; and at 56 days, when they are 63 days old. All length changes shall be calculated as percentage change, based on the effective gage length of the bar, assuming as unity the gage reading after the bar has been in the moist closet for 2 days and in fresh water for 5 days, immediately prior to being placed in a test solution. Previous to weighing the bars after exposure to the solutions, the bars shall be lightly brushed, using a 1-in. flat brush of steel wire bristles, with five full strokes lengthwise for each of the four sides of a bar. At no time shall other than loose material purposely be removed from a bar by brushing or otherwise. Immediately prior to weighing, the bars shall be wiped with a towel or absorbent paper to remove superficial water.

REPORT OF COMMITTEE C-5*
ON
FIRE TESTS OF MATERIALS AND CONSTRUCTION

Since the preceding report in June, 1945, Committee C-5 on Fire Tests of Materials and Construction has held one meeting on March 21, 1946, at Washington, D. C. Preceding this meeting and the meetings of subcommittees held in connection therewith, considerable progress was made by correspondence and meetings among members of groups concerned with specific projects. A meeting is scheduled for June 27, at Buffalo, N. Y.

The committee now consists of 49 members, of whom 21 are classified as producers, 9 as consumers, 15 as general interest members, and 4 as consulting members.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, S. H. Ingberg.
Vice-Chairman, A. L. Brown.
Secretary, Hugh M. Robinson.

I. NEW TENTATIVE

The committee recommends a new Method for Determination of Combustible Properties of Treated Wood by the Fire-Tube Method[1] for publication as tentative. This method, developed by the U. S. Forest Products Laboratory, has been in use for a sufficient period to enable accumulation of a considerable volume of data from its use in fire tests of wood of various species impregnated with a wide range in chemicals and resulting loadings. A simplified method and equipment is included as an alternative where only the final weight loss is required.

While covering the same field of application as the crib test method, it is deemed desirable to standardize both methods since convenience in sampling or availability of equipment may make one method or the other preferable for given conditions.

II. TENTATIVE REVISIONS OF STANDARDS

The committee recommends for publication as a tentative revision of the Standard Methods of Fire Tests of Building Construction and Materials (C 19 – 41)[2] the changes shown in the Appendix[3] to this report. The following is an explanation of the reasons for these several proposed changes:

Section 2, Time-Temperature Curve.—A closer definition of the time-temperature curve for control of fire tests is proposed by the addition of an appendix giving temperatures at 5-min. intervals up to 2 hr. and at 10-min. intervals from 2 to 8 hr., within which latter period the increase in temperature is linear with time. There is also given therein the area under the time-temperature curve, taken for Fahrenheit units to a base line of 68 F., and for Centigrade units to a base line of 20 C.

Section 5, Report of Results.—This section is amended to require reporting of results of fire-endurance tests to the nearest integral minute. A method is proposed for correcting the observed failure time in fire-endurance tests because of variation of the intensity of the furnace fire exposure from that prescribed as standard. The method of correction is based on the theory of heat flow in solid materials and has been verified experimentally to the extent the limited applicable data afford a basis.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] This method was accepted as tentative by the Society and appears in the 1946 Book of A.S.T.M. Standards, Part II.

[2] 1944 Book of A.S.T.M. Standards, Part II.

[3] See p. 297.

Section 21, Tests of Floors and Roofs.—Paragraph (*b*) of this section is modified to eliminate the requirement for the application of the excess load to roof constructions after the hose stream test. This is based on the assumption that substantially no loads other than the dead load would be present on roofs after a fire and hence the test for integrity by applying twice the live load on cooling after the hose stream test would be less representative than for floors.

Alternate Test for Steel Columns.—New sections are submitted describing a method of conducting fire tests of structural steel columns without application of load, the limit of fire endurance being defined by the temperatures attained in the column steel. For this type of column the relation between strength and temperature has been quite well established in full-scale tests as well as in tests of smaller specimens. It is expected that tests will be conducted during the coming year to obtain further information on the range of application of the method.

Tests of Ceiling Constructions.—New sections are submitted describing a method of test of ceiling constructions such as under an attic or other location where there is no floor or roof sheathing or slab resting on the members to which the ceiling is immediately attached. The criteria of performance are based on structural failure and the protection given combustible construction in contact with or adjacent to the upper face. Further criteria, such as the temperature of the upper face as indicating safety of ties and fastenings, have been considered but, in absence of further test data, no limits thereon can as yet be specified.

The degree of fire resistance of such ceilings is important on account of possibility of spread of fire through the spaces above them, often extending undivided over the whole or considerable portions of a building.

III. ADOPTION OF TENTATIVE METHOD AS STANDARD

Tentative Method of Test for Fire-Retardant Properties of Wood (C 160 - 41 T)[2], using the crib test, has been in this status for five years, during which time considerable experience has accumulated with its use. This indicated a need of clarification as to the purpose of the method, particularly of the sampling requirements, in view of the fact that it is in general not practical to treat structural members in the usual dimensions so approximately uniform loading of chemicals will obtain throughout the cross-section. The sampling paragraphs were accordingly modified to indicate that the present requirements apply for the condition of penetration of treating solution throughout the section, and generally applicable for trim and other stock in the smaller range of thickness. Except for a change in the title, rearrangement of paragraphs, and minor changes and additions, which are shown in the Appendix to this report, the proposed standard is otherwise the same as the present tentative method. The committee accordingly recommends that this method, as revised, be approved for reference to letter ballot of the Society for adoption as standard.

GENERAL ACTIVITIES OF COMMITTEE

Fire Tests for Wood Given Partial or Surface Treatment.—The committee recognizes the importance of a method for fire test of wood that has been given partial or surface treatment and has given it consideration to the point where the further steps needed appear fairly well defined. While one or both of the types of apparatus, fire tube and crib test equipment, can apparently be used for the purpose, different requirements for sampling will have to be applied than those for wood treated throughout the cross-section. Development of a method for obtaining representative samples from lots of treated material, often of large-size members, involves difficulties that it has not as yet been possible to fully resolve, in view of the limited amount of specific applicable information and the extent of consideration the subcommittee concerned has been able to give this project.

Fire Tests of Window Assemblies.—No standard covering methods of fire tests of window assemblies can as yet be submitted although progress has been made in reconciling conflicting viewpoints and clearing up misunderstandings that have impeded progress on this project. Considering the vulnerability of glass to high temperatures, a closer tolerance, within attainable limits, for furnace temperatures in fire test thereof than for fire tests of other constructions may be justified. Otherwise, the past experience from exposure in building fires and in fire tests may indicate the general line of applicable requirements.

Fire Tests of Acoustical and Similar Finishes.—The work during the year has been concerned largely with conducting tests and developing equipment and methods for determining the speed and extent of flame travel over the surface and the density of the smoke produced. Equipments of the flame-tunnel type have been indicated as possibly best adapted for these purposes.

Fire Tests of Roof Coverings.—Further progress has been made on this project, including tentative fire-resistance classifications of built-up and prepared roof coverings, based on performance in given types of tests. While no limits on performance or details of the pertaining testing methods have as yet been defined, what has been accomplished is a necessary preliminary thereto.

As a basis for the revisions and amendments to Standard Method C 19 – 41, further fire tests of ceiling constructions, sponsored by the National Housing Agency, were conducted at the National Bureau of Standards, which also gave data of value in the design of multiple dwellings. A number of fire tests of steel roof deck constructions were also conducted, sponsored by members of the steel roof deck industry, from which information was obtained on what differentiation, if any, should be made between testing methods and end-point criteria for roof constructions as compared with those for floors.

Subcommittee I on Fire Tests of Materials and Construction (G. N. Thompson, chairman) and its groups have been active throughout the year in developing the revisions to the general fire test specifications that are proposed. Subcommittee II on Fire Tests of Lumber (W. J. Krefeld, chairman) has likewise made good progress in clearing up matters that have been on its agenda for several years. With completion of projects now in progress, the standards with which it has been concerned will cover in large part the requirements for the various categories that need be recognized in fire test methods for treated wood.

In accordance with the action of the Executive Committee of the Society, methods of conducting fire tests proposed by other committees will be referred to Committee C-5 for comment in the interest of obtaining uniformity in procedures so far as is consistent with the purposes and materials for which the individual types of tests are developed.

The recommendations appearing in this report have been submitted to the letter ballot of the committee, which consists of 45 voting members; 35 members returned their ballots with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE METHOD			
Test for Combustible Properties of Treated Wood by the Fire-Tube Method	30	1	4
II. TENTATIVE REVISION OF STANDARD			
Fire Tests of Building Construction and Materials (C 19 - 41)..........................	27	1	7
III. ADOPTION OF TENTATIVE AS STANDARD			
Method of Test for Combustible Properties of Treated Wood by the Crib Test Method (C 160 - 41 T)......................	30	0	5

This report has been submitted to the letter ballot of the committee, which consists of 49 members; 38 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

S. H. INGBERG,
Chairman.

H. M. ROBINSON,
Secretary.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR FIRE TESTS OF MATERIALS AND CONSTRUCTION

In this Appendix are given proposed revisions in a tentative method and a proposed tentative revision of a standard, which are referred to earlier in this report.[1] The existing tentative and standard appear in their present form in the 1944 Book of A.S.T.M. Standards, Part II, pp. 1259 and 234, respectively.

REVISION AND ADOPTION OF TENTATIVE AS STANDARD

Tentative Method of Test for Fire Retardant Properties of Wood (C 160 - 41 T):[2]

Title.—Change title to read as follows by addition of italicized words and omission of those in brackets: "Tentative Method of Test for [Fire-Retardant] *Combustible* Properties of *Treated* Wood *by the Crib Test Method.*"

Section 1.—Change the statement of scope to read as follows:

> 1. This method of test covers a procedure for fire tests of wood treated to reduce flammability. The test relates to properties of treated wood as such, rather than to the performance of a fabrication used as an element of construction. Performance under this test shall be as prescribed in requirements applicable to materials intended for specific uses.

Section 3.—Renumber as Section 2 without change in text.

New Section 3.—Include new introductory paragraph and include and amend parts of present Sections 2 and 4 to read as follows:

> 3. *Sampling and Test Specimens.*—For interior trim or other lumber requiring penetration of treating solution throughout the section but not for wood given incomplete penetration, the specimens shall be prepared as follows:

Paragraph 3. (a).—Same as Paragraph 2(*a*) except omit first sentence and modify the second sentence to read as follows by addition of italicized words and the omission of those in brackets:

> [The shipment shall be carefully examined and a representative sample selected.] One sample shall be taken from *the lot for* each 5000 board feet or fraction thereof.

Omit present Sections 2(*b*) and 2(*c*).

Paragraph 3. (b).—Same as Paragraph 4(*a*) with additions of italicized words to read as follows:

> (*b*) The *crib test* specimen *cut from the selected sample* shall consist of 24 pieces $\frac{1}{2}$ by $\frac{1}{2}$ in. in cross-section and 3 in. in length, with surfaces smooth-sawed to dimensions within plus or minus $\frac{1}{32}$ in. The specimen shall be representative of the entire cross-section of the sample.

Paragraphs 3(c) to (f).—Same as Paragraphs 4(*b*) to (*e*).

Section 5.—Renumber as Section 4 and change "moisture specimen" to "moisture test specimen" wherever it appears.

Section 6.—Renumber as Section 5 and add the following at the end of Paragraph (*b*): "A gas pressure regulator is desirable."

Section 7.—Renumber as Section 6.

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[1] See p. 293.

[2] 1944 Book of A.S.T.M Standards, Part II.

Standard Methods of Fire Tests of Building Construction and Materials (C 19 – 41):[2]

Section 2.—Add the following at the end of Section 2 after the time-temperature table: "For a closer definition of the time-temperature curve see the Appendix."

Section 5.—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

5. (*a*) Results shall be reported in accordance with the performance in the tests prescribed in these specifications. They shall be expressed in time periods of resistance [as for example 4 hr., $\frac{1}{2}$ hr., etc.], *to the nearest integral minute.*

Add the following new Paragraph (*b*):

(*b*) Where the indicated resistance period is $\frac{1}{2}$ hr. or over and determined by the average or maximum temperature rise on the unexposed surface or within the test sample, or by failure under load, a correction shall be applied for variation of the furnace exposure from that prescribed by multiplying the indicated period by two thirds of the difference in area between the curve of average furnace temperature and the standard curve for the first three fourths of the period and dividing the product by the area between the standard curve and a base line of 68 F. (20 C.) for the same part of the indicated period, the latter area increased by 54 Fahr.-hr. or 30 Cent.-hr. (3240 Fahr.-min. or 1800 Cent.-min.), to compensate for the thermal lag of the furnace thermocouples during the first part of the test. For fire exposure in the test higher than standard, the indicated resistance period shall be increased by the amount of the correction and be similarly decreased for fire exposure below standard (Note).

NOTE.—The correction can be expressed by the following formula:

$$C = \frac{2I(A - A_s)}{3(A_s + L)}$$

where:

C = correction in the same units as I,

I = indicated fire-resistance period,

A = area under the curve of indicated average furnace temperature for the first three quarters of the indicated period,

A_s = area under the standard furnace curve for the same part of the indicated period, and

L = lag correction in the same units as A and A_s.

New Sections.—Add the following new Sections 19 to 23, renumbering the subsequent sections accordingly:

ALTERNATE TEST FOR STRUCTURAL STEEL COLUMNS

19. *General.*—This test procedure does not require column loading at any time and may be used at the discretion of the testing laboratory to evaluate steel column protections that are not required by design to carry any of the column load.

20. *Size and Character of Sample.*—(*a*) The size of the steel column used shall be such as to provide a test specimen which is truly representative of the design, materials and workmanship for which classification is desired. The protection shall be applied according to the methods of acceptable field practice. The length of the protected column shall be at least 8 ft. The column shall be vertical during application of the protection and during the fire exposure.

(*b*) The applied protection shall be restrained against longitudinal temperature expansion greater than that of the steel column by rigid steel plates or reinforced concrete attached to the ends of the steel column before the protection is applied. The size of the plates or amount of concrete shall be adequate to provide direct bearing for the entire transverse area of the protection.

(*c*) The ends of the specimen, including the means for restrainst shall be given sufficient thermal insulation to prevent appreciable direct heat transfer from the furnace.

21. *Temperature Measurement.*—The temperature of the steel in the column shall be measured by at least three thermocouples located at each of four levels. The upper and lower levels shall be 2 ft. from the ends of the steel column, and the other two intermediate levels shall be equally spaced. The thermocouples at each level shall be so placed as to measure significant temperatures of the component elements of the steel section.

22. *Exposure to Fire.*—During the fire endurance test the specimen shall be exposed to fire on all sides for its full length.

23. *Conditions of Acceptance.*—The test shall be regarded as successful if the transmission of heat through the protection during the period of fire exposure for which classification is desired does not raise the average (arithmetical) temperature of the steel at any one of the four levels above 1000 F., or does not raise the temperature above 1200 F. at any one of the measured points

Section 21 (*b*).—Change to read as follows by the addition of the italicized

TABLE I.—STANDARD TIME-TEMPERATURE CURVE FOR CONTROL OF FIRE TESTS.

Time	Temperature, deg. Fahr.	Area Above 68 F. Base		Temperature, deg. Cent.	Area Above 20 C. Base	
hr.:min.		deg. Fahr.-min.	deg. Fahr.-hr.		deg. Cent.-min.	deg. Cent.-hr.
0:00	68	00	0	20	00	0
0:05	1 000	2 330	39	538	1 290	22
0:10	1 300	7 740	129	704	4 300	72
0:15	1 399	14 150	236	760	7 860	131
0:20	1 462	20 970	350	795	11 650	194
0:25	1 510	28 050	468	821	15 590	260
0:30	1 550	35 360	589	843	19 650	328
0:35	1 584	42 860	714	862	23 810	397
0:40	1 613	50 510	842	878	28 060	468
0:45	1 638	58 300	971	892	32 390	540
0:50	1 661	66 200	1 103	905	36 780	613
0:55	1 681	74 220	1 237	916	41 230	687
1:00	1 700	82 330	1 372	927	45 740	762
1:05	1 718	90 540	1 509	937	50 300	838
1:10	1 735	98 830	1 647	946	54 910	915
1:15	1 750	107 200	1 787	955	59 560	993
1:20	1 765	115 650	1 928	963	64 250	1 071
1:25	1 779	124 180	2 070	971	68 990	1 150
1:30	1 792	132 760	2 213	978	73 760	1 229
1:35	1 804	141 420	2 357	985	78 560	1 309
1:40	1 815	150 120	2 502	991	83 400	1 390
1:45	1 826	158 890	2 648	996	88 280	1 471
1:50	1 835	167 700	2 795	1 001	93 170	1 553
1:55	1 843	176 550	2 942	1 006	98 080	1 635
2:00	1 850	185 440	3 091	1 010	103 020	1 717
2:10	1 862	203 330	3 389	1 017	112 960	1 882
2:20	1 875	221 330	3 689	1 024	122 960	2 049
2:30	1 888	239 470	3 991	1 031	133 040	2 217
2:40	1 900	257 720	4 295	1 038	143 180	2 386
2:50	1 912	276 110	4 602	1 045	153 390	2 556
3:00	1 925	294 610	4 910	1 052	163 670	2 728
3:10	1 938	313 250	5 221	1 059	174 030	2 900
3:20	1 950	332 000	5 533	1 066	184 450	3 074
3:30	1 962	350 890	5 848	1 07	194 940	3 249
3:40	1 975	369 890	6 165	1 079	205 500	3 425
3:50	1 988	389 030	6 484	1 086	216 130	3 602
4:00	2 000	408 280	6 805	1 093	226 820	3 780
4:10	2 012	427 670	7 128	1 100	237 590	3 960
4:20	2 025	447 180	7 453	1 107	248 430	4 140
4:30	2 038	466 810	7 780	1 114	259 340	4 322
4:40	2 050	486 560	8 110	1 121	270 310	4 505
4:50	2 062	506 450	8 441	1 128	281 360	4 689
5:00	2 075	526 450	8 774	1 135	292 470	4 874
5:10	2 088	546 580	9 110	1 142	303 660	5 061
5:20	2 100	566 840	9 447	1 149	314 910	5 248
5:30	2 112	587 220	9 787	1 156	326 240	5 437
5:40	2 125	607 730	10 129	1 163	337 630	5 627
5:50	2 138	628 360	10 473	1 170	349 090	5 818
6:00	2 150	649 120	10 819	1 177	360 620	6 010
6:10	2 162	670 000	11 167	1 184	372 230	6 204
6:20	2 175	691 010	11 517	1 191	383 900	6 398
6:30	2 188	712 140	11 869	1 198	395 640	6 594
6:40	2 200	733 400	12 223	1 204	407 450	6 791
6:50	2 212	754 780	12 580	1 211	419 330	6 989
7:00	2 225	776 290	12 938	1 218	431 270	7 188
7:10	2 238	797 920	13 299	1 225	443 290	7 388
7:20	2 250	819 680	13 661	1 232	455 380	7 590
7:30	2 262	841 560	14 026	1 239	467 540	7 792
7:40	2 275	863 570	14 393	1 246	479 760	7 996
7:50	2 288	885 700	14 762	1 253	492 060	8 201
8:00	2 300	907 960	15 133	1 260	504 420	8 407

words and the omission of the word in brackets:

(*b*) The construction shall have sustained the applied load during the fire and hose stream test as specified in Section 9, without passage of flame, of gases hot enough to ignite cotton waste, or of the hose stream [and]. After cooling, but within 72 hr. after completion of the hose stream test, *floor specimens* shall sustain a total load equal to the dead load plus twice the superimposed load specified above.

New Sections.—Add the following new Sections 27 to 29, renumbering the subsequent sections accordingly:

TESTS OF CEILING CONSTRUCTIONS

27. *Size of Sample.*—The area exposed to fire shall not be less than 180 sq. ft. with neither dimension less than 12 ft. and the ceiling surface at its edges shall be in contact with the test furnace structure.

28. *Test Sample and Enclosure.*—The test ceiling construction shall include all the structural members and details including hangers, if any, but shall not include walkways. Above the ceiling during the test, shall be provided a tight enclosing construction with flat roof placed with the lower edge of its supports not more than 30 in. above the ceiling joists. The top of the enclosure shall be made of cement-asbestos board $\frac{1}{4}$ in. in thickness under asbestos millboard $\frac{1}{2}$ in. in thickness, and the side walls of 8-in. common brick, or it shall be of a construction having equivalent heat conductivity and heat capacity. Where use of the ceiling under a combustible construction is contemplated, at least five 1-in. pine boards, each having an area of 2 sq. ft. shall be attached to the top of the enclosure under which the exposed surface temperatures can be measured.

29. *Conditions of Acceptance.*—The test shall not be regarded as successful unless the following conditions are met:

(*a*) The ceiling shall have withstood the fire endurance test without the passage of flame or ignition of combustible members or materials forming part of the construction above the ceiling as evidenced by glow or flame.

(*b*) Transmission of heat through the ceiling during the fire endurance test shall not have been such as to raise the average temperature above the test ceiling more than indicated in the following items (*1*), (*2*) and (*3*). The limiting temperatures shall be the average of those taken at not less than five points, one of which shall be approximately at the center, and four at approximately the centers of the quarter sections.

(*1*) With combustible supports or other combustible material in contact with the ceiling, the temperature increase at the points of contact shall not exceed 250 F.

(*2*) With combustible supports or other combustible material not in contact with the ceiling, the temperature increase on the surface of any combustible members or combustible material adjacent to the ceiling shall not exceed 250 F. The temperature on the exposed surface of combustible members not in contact with the ceiling shall be measured under a sheet of mica approximately 0.002 in. in thickness.

(*3*) With no combustible material above the ceiling construction, the average temperature measured on the lower surface of the main structural supporting members (beams or slabs) shall not exceed 1200 F. and the average temperature of the top and bottom of the beams, when used, shall not exceed 1000 F.

Appendix.—Add as an Appendix the accompanying Table I.

REPORT OF COMMITTEE C-7*

ON

LIME

Committee C-7 on Lime did not hold a meeting during the year, but a meeting of the Advisory Subcommittee was held in Boston, Mass., on April 23, 1946.

The total membership at present is 47, of whom 24 are classified as producers, 5 as consumers, and 18 as general interest members. It is with regret that we note the death of one of our active members and vice-chairman, J. F. Carle of the Southern Testing Laboratories, on December 16, 1945.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, W. C. Voss.
Vice-Chairman, J. A. Murray.
Vice-Chairman, L. K. Herndon.
Secretary, G. J. Fink.

STANDARDS SIX YEARS WITHOUT REVISION

The following standards have been published for six years without revision and it is the recommendation of Committee C-7 that they be continued as standard:

Standard Specifications for:

Quicklime for Structural Purposes (C 5 – 26),
Sand for Use in Plaster (C 35 – 39),
Quicklime and Hydrated Lime for Cooking of Rags in Paper Manufacture (C 45 – 25),
Quicklime for Sulfite Pulp Manufacture (C 46 – 27),
Hydrated Lime for Varnish Manufacture (C 47 – 27),
Quicklime and Hydrated Lime for Use in the Textile Industry (C 48 – 24),
Quicklime and Hydrated Lime for Water Treatment (C 53 – 39), and

Standard Methods of:

Sampling, Inspection, Packing and Marking of Quicklime and Lime Products (C 50 – 27).

Three specifications for lime and lime products have been sent to letter ballot this year after considerable work in Subcommittee II on Structural Lime. These specifications include Proposed Tentative Specifications for Hydrated Lime for Masonry Purposes, Proposed Tentative Specifications for Special Finishing Hydrated Lime, and a proposed tentative revision of the Standard Specifications for Normal Finishing Hydrated Lime (C 6 – 44). These are being held for discussion at the Buffalo meeting.[1]

The Advisory Committee held one meeting during the year at Boston on April 23, 1946, at which the program for the coming year was discussed. It was decided to enforce the requirements of the Committee Regulations as to attendance at meetings and all members have been notified to this effect. Recommendations regarding membership will be presented at the Buffalo meeting.[1]

Subcommittee III on Lime for Chemical Industries (R. W. McAllister, chairman) has circulated a proposed set of master specifications to the committee for comment. The replies will be discussed at Buffalo.

This report has been submitted to letter ballot of the committee, which consists of 53 members; 28 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. C. Voss,
Chairman.

L. K. Herndon,
Secretary.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] These specifications were accepted as tentative by the Society and appear in the 1946 Book of A.S.T.M. Standards, Part II.

REPORT OF COMMITTEE C-8*

ON

REFRACTORIES

Committee C-8 on Refractories held one meeting during the year, at Battelle Memorial Institute, Columbus, Ohio, on November 20, 1945.

Three new members were elected to the committee during the year: L. A. Smith, representing Jones & Laughlin Steel Corp.; R. P. Stevens, representing Chicago Retort and Fire Brick Co.; and J. L. Carruthers, representing Edward Orton Jr. Ceramic Foundation. L. C. Hewitt and S. M. Phelps were appointed to fill the vacancies created on the Advisory Board and the chairmanship of the Subcommittee on Tests, respectively.

The committee records with sorrow the death in April, 1945, of F. A. Harvey, who was a member of the A.S.T.M. for over 25 years and a very active member of Committee C-8. Mr. Harvey was a most valuable contributor to the work of the committee by his unceasing efforts to promote the activities of the committee through his technical knowledge and good judgment.

The committee also regrets the loss of another member, Dean M. E. Holmes, who passed away in May, 1946. He was a member of Committee C-8 for 19 years, and served for many years as chairman of the Subcommittee on Nomenclature.

As the committee did not hold a spring meeting this year, the election of officers for the ensuing term of two years was conducted by letter ballot vote of the membership of the committee, resulting in the selection of the following:

Chairman, J. D. Sullivan.
Vice-Chairman, L. J. Trostel.
Secretary, S. M. Phelps.

I. New Standard, Immediate Adoption

After its revision in 1942, a number of suggestions were made for the improvement of the Standard Methods of Testing Insulating Fire Brick (Compressive Strength, Flexural Strength, and Permanent Linear Change After Heating) (C 93 – 42).[1] The principal change proposed is the separation of the test for permanent linear change after heating from the other two tests, and its presentation as a separate new standard similar to Standard Method of Test for Permanent Linear Change After Reheating of Refractory Brick (C 113 – 36). The proposed new standard incorporates a heating schedule and states specifically the permissible temperature variation, the acceptable method for measuring the temperature, the position of the test specimens in the furnace, and other details.

Because this subject matter is preferred as standard rather than tentative, and because the new presentation does not incorporate matter that is essentially new or different, Committee C-8 recommends the immediate adoption of the proposed Standard Method of Test for Permanent Linear Change Upon Reheating of Insulating Fire Brick[2] and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that this proposed standard may be referred to letter ballot of the Society.

*** Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.**

[1] 1944 Book of A.S.T.M. Standards, Part II.

[2] This method was accepted as standard by the Society and appears in the 1946 Book of A.S.T.M. Standards Part II.

II. REVISION OF STANDARD, IMMEDIATE ADOPTION

Standard Methods of Testing Insulating Fire Brick (Compressive Strength, Flexural Strength, and Permanent Linear Change After Heating) (**C 93 – 42**).[1]—In order to bring these methods of test more in line with other C-8 methods, the committee recommends separation of the test for permanent linear change after heating, retaining in the original standard the two mechanical methods of testing. The committee further recommends the use of the terms "cold crushing strength" and "modulus of rupture" in place of "compressive strength" and "flexural strength," to conform to usage in the Standard Methods of Test for Cold Crushing Strength and Modulus of Rupture of Refractory Brick and Shapes (C 133 – 39). The designation of a required sensitivity for the testing machine is also recommended. Accordingly, Committee C-8 recommends that the proposed Standard Methods of Test for Crushing Strength and Modulus of Rupture of Insulating Fire Brick at Room Temperature (C 93 – 46)[3] be accepted as a revision of the present standard method (C 93 – 42) and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that this revision may be referred to letter ballot of the Society.

Standard Method of Test for Permanent Linear Change After Reheating of Refractory Brick (**C 113 – 36**).[1]—This standard method has been on the books of the Society for over six years without revision. The committee recommends that it be revised to cover the testing of fireclay plastic refractories as specified in the Tentative Method of Test for Combined Drying and Firing Shrinkage of Fireclay Plastic Refractories (C 179 – 43 T). The committee also recommends the use of brick specimens 3 in. in thickness, in addition to the $2\frac{1}{2}$-in. size, and proposes the inclusion in the standard of some reference to a suitable type of testing furnace. The committee therefore recommends revision of this standard as follows:

Title.—Change "Refractory Brick" to read "Refractories."

Section 2.—Add a new footnote 3 to this section to read as follows, renumbering the subsequent footnotes accordingly: "One kiln design suitable for making a reheat test is described in the *Bulletin* of the American Ceramic Society, Vol. 19, p. 336 (1940)."

Section 3 (a).—Change this section to read as follows: "Three test specimens shall be used, and these shall measure 9 by $4\frac{1}{2}$ by either $2\frac{1}{2}$ or 3 in. These may be commercial brick or test pieces of that size cut out of larger shapes or, if fireclay plastic refractories, three molded specimens of those dimensions."

Since these changes are of a minor nature, the committee asks for a nine-tenths affirmative vote at the Annual Meeting in order that this revision may be referred to letter ballot of the Society. The method in general, however, is reaffirmed by the committee.

Standard Methods of Test for Apparent Porosity, Water Absorption, Apparent Specific Gravity, and Bulk Density of Burned Refractory Products (**C 20 – 41**).[1]—A new vacuum method for determining porosity has been called to the attention of the committee. This method is in agreement with the C-8 boiling method when conducted on samples of representative fire clay, but is in disagreement with tests on bars made of raw clay. The committee recommends the following changes in Methods C 20, thereby limiting this porosity test to fireclay brick:

Title.—Change the word "Products" to read "Brick."

Section 1.—In line 3, change the word "products" to read "brick."

In the note at the end of this section change the word "materials" to read "refractories."

The committee accordingly recommends that this revision be approved and asks for a nine-tenths affirmative vote at the Annual Meeting in order that this recommendation may be referred to letter ballot of the Society.

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[3] These revised methods were accepted by the Society and appear in the 1946 Book of A.S.T.M. Standards Part II.

III. ADOPTION OF TENTATIVES AS STANDARD

Since no adverse criticisms of the following tentative methods have been received, the committee recommends that they be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Methods of:[1]

Test for Sieve Analysis and Water Content of Refractory Materials (C 92 - 43 T), and
Test for Combined Drying and Firing Shrinkage of Fireclay Plastic Refractories (C 179 - 43 T).

Tentative Symbols for:[1]

Heat Transmission (C 108 - 44 T).

IV. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARD

Standard Method of Test for Pyrometric Cone Equivalent (P.C.E.) of Refractory Materials (**C 24 - 42**).[1]—The tentative revision[1] of this method, published in 1944, consisted of the inclusion of a drawing of the excess length type of split mold for the pyrometric test cone. The committee recommends that this tentative revision be approved for reference to letter ballot of the Society for adoption as standard.

Standard Definitions of Terms Relating to Refractories (**C 71 - 45**).[4]—The definition of the term "calcining of refractory materials" was published in 1942 as a tentative revision[4] of this standard. Since no adverse criticism has been received, the committee recommends that this definition be approved for reference to letter ballot of the Society for adoption as standard.

V. TENTATIVE REVISIONS OF STANDARDS

Standard Methods of Panel Test for Resistance to Thermal and Structural Spalling of Refractory Brick (**C 38 - 45**),[4] *Panel Test for Resistance to Thermal and Structural Spalling of High Heat Duty Fireclay Brick* (**C 107 - 45**),[4] and *Panel Test for Resistance to Thermal and Structural Spalling of Super Duty Fireclay Brick* (**C 122 - 45**).[4]—The manner in which the spalls are removed from the brick on completion of the panel test is not described in the present standards. The committee recommends the following paragraph for publication as a tentative revision to replace the present Section 7 of Standard Method C 38 and Section 5 (*f*) of Standard Methods C 107 and C 122:

> When the test panel has cooled sufficiently so that the brick can be handled, the panel shall be carefully dismantled. Each test specimen shall be cleaned to free it of the joint-forming kaolin and in such a manner as to remove spalls. This shall be carried out by the use of an 8 or 9-in. brick trowel which is handled so as to provide a brisk scraping action without impacts. The movement of the trowel shall be directed toward and beyond the spalled face of the brick. This operation shall be carried out on the two 4½ by 9-in. faces and the two ends of the brick until no additional spalls are removed. No attempt shall be made to separate pieces that might be removed by prying or tapping. The brick shall be reweighed to the nearest 0.05 lb. and the loss in weight recorded as percentage of the original weight.

Standard Specifications for Refractories for Malleable Iron Furnaces with Removable Bungs and for Annealing Ovens (**C 63 - 41**).[1]—Heretofore, these specifications have failed to indicate the number of specimens required for each test, and the manner of their selection. In order to guide the consumer in sending sufficient and proper specimens to a laboratory for testing, the committee recommends the following for publication as a tentative revision:

New Section.—Add the following as a new Section 9, renumbering the subsequent sections accordingly:

> 9. ***Samples for Test.***—The number of brick required for testing the various types of fireclay

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[4] 1945 Supplement to Book of A.S.T.M. Standards, Part II.

brick referred to in these specifications is given in the accompanying Table I. The test brick shall be 9 by 4½ by 2½ in. or specimens of that size cut from larger shapes, utilizing as far as possible existing plane surfaces. Shapes of suitable size may be submitted provided the laboratory has facilities for cutting 9-in. brick from them. The test specimens shall be selected at random from each shipment of 50,000 brick or less.

Standard Method of Panel Test for Resistance to Thermal and Structural Spalling of Fireclay Plastic Refractories (**C 180 - 45**).[4]—In order to provide a detailed description for removal of the spalls after completion of the panel test, the committee recommends the following paragraph for publication as a tentative revision:

TABLE I.—NUMBER OF BRICK REQUIRED FOR TESTING.

Test Methods to be Used	Types of Brick and Number Required for Testing				
	H	I	J	K	L
Pyrometric cone equivalent	1	1	1	1	1
Load test	2	..	..	2	..
Permanent linear change	6	..	6	6	6
Panel spalling	..	14	..	..	..
Modulus of rupture	..	5	..	..	..
Extra brick[a]	1	2	1	1	1
Total without brick for size variation	10	22	8	10	8
Total when size variation is to be obtained by laboratory[b]	22	22	22	22	22

[a] Extra brick are included so as to provide for those which may become damaged or broken in shipment.

[b] Twenty brick are required for size variation but the measurements are made on the test brick before they are used for any of the other tests.

Section 6 (f).—Change to read as follows:

> (*f*) When the test panel has cooled sufficiently so that the brick can be handled, the panel shall be carefully dismantled. Each test specimen shall be cleaned by removing the joint-forming kaolin on the two 4½ by 9-in. faces and the two ends. This shall be carried out by the use of an 8 or 9-in. brick trowel which is handled so as to provide a brisk scraping action without impacts. The movement of the trowel shall be directed away from the spalled face of the brick so as not to remove spalls while scraping off the kaolin. After this operation each test brick, and any spalls which may have unavoidably been removed, shall be weighed to the nearest 0.05 lb. The spalls shall then be removed from the brick by making use of the trowel in the manner described, with the exception that the scraping shall be directed toward and beyond the spalled face of the brick. This operation shall be carried out on the two 4½ by 9-in. faces and the two ends of the brick until no additional spalls are removed. No attempt shall be made to separate pieces that might be removed by prying or tapping. The brick shall be reweighed to the nearest 0.05 lb. which provides the information for calculation of the spalling loss.

TABLE II.—NUMBER OF BRICK REQUIRED FOR TESTING.

Test Methods to be Used	Types of Brick and Number Required for Testing						
	A	B	C	D	E	F	G
Pyrometric cone equivalent	1	1	1	1	1	1	1
Load test	2	2	2	..	2	2	..
Permanent linear change	6	6	6	6	..	..	..
Panel spalling	14	14	..	14	..	..	..
Modulus of rupture	..	..	..	..	..	5	5
Water absorption	..	..	5	..	..	..	..
Extra brick[a]	2	2	2	2	1	1	1
Total without brick for size variation	25	25	16	23	4	9	7
Total when size variation is to be obtained by laboratory[b]	25	25	22	23	22	22	22

[a] Extra brick are included so as to provide for those which may become damaged or broken in shipment.

[b] Twenty brick are required for size variation but the measurements are made on the test brick before they are used for any of the other tests.

Standard Specifications for Refractories for Heavy Duty Stationary Boiler Service (**C 64 - 41**).[1]—As a guide to the consumer in determining the number of specimens desired and the manner of their selection for each of the test methods referred to in these specifications, the committee recommends the following for publication as a tentative revision:

New Section.—Add the following as a new Section 7, renumbering the subsequent sections accordingly:

> 7. *Samples for Test.*—The number of brick required for testing the various types of fireclay brick referred to in these specifications is given in the accompanying Table II. The test brick shall be 9 by 4½ by 2½ in.[a] or specimens of that size cut from larger shapes, utilizing as far as

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[a] Test brick 3 in. in thickness may be used for the permanent linear change and spalling tests.

possible existing plane surfaces. Shapes of suitable size may be submitted provided the laboratory has facilities for cutting 9-in. brick from them. The test specimens shall be selected at random from each shipment of 50,000 brick or less.

Standard Specifications for Refractories for Incinerators (**C 106 – 41**).[1]—To provide instructions for sending sufficient and proper specimens to be tested by the various standard methods listed in these specifications, the committee recommends the following for publication as a tentative revision:

TABLE III.—NUMBER OF BRICK REQUIRED FOR TESTING.

Test Methods to be Used	Types of Brick and Number Required for Testing		
	A	F	G
Pyrometric cone equivalent	1	1	1
Load test	2	2	..
Permanent linear change	6	..	..
Panel spalling	14	...	..
Modulus of rupture	..	5	5
Extra brick[a]	2	1	1
Total without brick for size variation.	25	9	7
Total when size variation is to be obtained by laboratory[b]	25	22	22

[a] Extra brick are included so as to provide for those which may become damaged or broken in shipment.

[b] Twenty brick are required for size variation but the measurements are made on the test brick before they are used for any of the other tests.

New Section.—Add the following as a new Section 6, renumbering the subsequent sections accordingly:

6. *Samples for Test.*—The number of brick required for testing the various types of fireclay brick referred to in these specifications is given in the accompanying Table III. The test brick shall be 9 by 4½ by 2½ in.[a] or specimens of that size cut from larger shapes, utilizing as far as possible existing plane surfaces. Shapes of suitable size may be submitted provided the laboratory has facilities for cutting 9-in. brick from them. The test specimens shall be selected at random from each shipment of 50,000 brick or less.

[a] Test brick 3 in. in thickness may be used for the permanent linear change and spalling tests.

Standard Specifications for Refractories for Moderate Duty Stationary Boiler Service (**C 153 – 41**).[1]—In order to indicate the number of specimens required for the several test methods referred to in these specifications, and the conditions of their selection, the committee recommends the following for publication as a tentative revision:

New Section.—Add the following as a new Section 6, renumbering the subsequent sections accordingly:

6. *Samples for Test.*—The number of brick required for testing the various types of fireclay brick referred to in these specifications is given in the accompanying Table IV. The test brick shall be 9 by 4½ by 2½ in.[a] or specimens of that size cut from larger shapes, utilizing as far as possible existing plane surfaces. Shapes of suitable size may be submitted provided the laboratory has facilities for cutting 9-in. brick from them. The test specimens shall be selected at random from each shipment of 50,000 brick or less.

TABLE IV.—NUMBER OF BRICK REQUIRED FOR TESTING.

Test Methods to be Used	Types of Brick and Number Required for Testing	
	H	G
Pyrometric cone equivalent	1	1
Load test	2	..
Permanent linear change	6	..
Modulus of rupture	..	5
Extra brick[a]	1	1
Total without brick for size variation..	10	7
Total when size variation is to be obtained by laboratory[b]	22	22

[a] Extra brick are included so as to provide for those which may become damaged or broken in shipment.

[b] Twenty brick are required for size variation but the measurements are made on the test brick before they are used for any of the other tests.

[a] Test brick 3 in. in thickness may be used for the permanent linear change test.

Standard Definitions of Terms Relating to Refractories (**C 71 – 45**).[4]—Dissatisfaction was expressed with the present standard definition for "fire clay" and the tentative definition for "air-setting refractory mortar." The following new definitions are offered in lieu of the present ones:

Fire Clay.—An earthy or stony mineral aggregate consisting essentially of hydrous silicates of aluminum with a low content of fluxes, plastic when sufficiently pulverized and wetted, rigid when dry, and possessing adequate strength when fired at a sufficiently high temperature.

Air-Setting Refractory Mortar.—A composition of finely ground materials, marketed in either a wet or dry condition, which may require tempering with water to attain the desired consistency and which is suitable for laying refractory brick and bonding them strongly upon drying and upon subsequent heating at furnace temperatures.

The committee recommends that these two new definitions be published as tentative revisions of Standard C 71.

Standard Method of Test for True Specific Gravity of Burned Refractory Materials (C 135 - 40).[1]—Several changes are recommended for the improvement of this standard. Because the method is applicable to unburned as well as to burned products, the word "burned" should be deleted from the title. No provision is made in this standard for the testing of samples in a pulverized condition. A source of error lies in the possible variation in temperature of the water-filled pycnometer when weighed with and without the sample. Any difference in temperature greater than plus or minus 0.5 F. (0.3 C.) in these two weighings is found to introduce an appreciable error in the true specific gravity reported. The inclusion of a boiling procedure as a permissible alternate method for removal of entrapped air from the pycnometer is recommended.

The committee therefore recommends the following for publication as a tentative revision of Standard Method C 135:

Title.—Delete the word "Burned."

Section 3.—Change the heading from "Test Specimens" to "Preparation of Sample;" also change this section to read as follows:

3. (*a*) Two pieces of walnut size shall be taken from different positions in a solid specimen in such a way as to exclude any part of the original exterior surface (skin surface). When an average value of specific gravity is desired, a composite sample shall be prepared by taking pieces in this manner from at least five different specimens.

(*b*) The pieces shall be crushed between hardened steel surfaces to a maximum particle size of $\frac{1}{8}$ in. The crushed material shall be thoroughly mixed and reduced by quartering to a test sample of 50 g.

(*c*) If the material submitted for test is already crushed or ground, a representative portion of at least 500 g. shall be thoroughly mixed and reduced by quartering to a test sample of 50 g.

(*d*) The entire 50-g. sample shall be ground in an agate mortar to such fineness that it will pass a No. 100 (149-micron) A.S.T.M. sieve[4] (equivalent to 100 mesh of the Tyler standard series). The grinding may be done by hand or by means of a mechanical sample grinder[3] so constructed as to prevent the introduction of any impurity. Any magnetic material introduced in the crushing or grinding shall be removed by a magnet. Care shall be taken in all stages of preparation of the test sample not to exclude any portions which are difficult to grind and to avoid any selective sampling.

Section 4(c).—Change the second sentence of this section to read as follows:

The pycnometer shall next be filled to one-fourth to one-half its capacity with distilled water, and the water boiled (Note 1) at atmospheric or under reduced pressure, for approximately 10 to 15 min. After boiling, the pycnometer shall be filled with distilled water (Note 2), cooled to room temperature t in a water bath, the stopper inserted, excess water from the stopper wiped off, and the pycnometer thoroughly dried with a lintless towel. The pycnometer and contents shall then be weighed and the contents reported as W_2.

Note 1.—Caution should be exercised during the boiling so that it is not sufficiently vigorous to cause loss of the sample due to popping. If the boiling is done at atmospheric pressure, it is advisable to insert with the stopper a thin strip of paper before the boiling operation. If reduced pressure is preferred, a suitable method of evacuation is described in Section 4(*e*) of the Methods of Test for Specific Gravity of Pigments (A.S.T.M. Designation: D 153).

Note 2.—The pycnometer should be filled so that there is an overflow of water through the capillary tube when the stopper is inserted. When wiping the excess water from the tip of the stopper, it must be done so as not to withdraw any water from the capillary tube. Any variation in room temperature t when obtaining weights W_1 and W_2 will introduce an appreciable error. Therefore, they should be obtained at the same temperature within the limits of plus or minus 0.5 F. (0.3 C.). The use of a constant temperature bath is desirable.

Standard Specifications for Fireclay Plastic Refractories for Boiler and Incinerator Services **(C 176 - 44)**.[1]—In order to indicate in these specifications the size of the sample of fireclay plastic refractory required for testing, and the method of shipment to the testing laboratory, the committee recommends the following for publication as a tentative revision:

Section 4.—Add the following as a new Section 4, renumbering the subsequent sections accordingly:

> 4. A sample of not less than 200 lb. shall be taken at random from each carload or less than carload shipment. If packed in moistureproof cartons of about 1-cu. ft. capacity, two cartons selected at random will suffice; if packed in steel drums, one drum of 200 lb. shall be selected.

Standard Method of Test for Workability Index of Fireclay Plastic Refractories **(C 181 - 45)**.[4]—No mention is made in this test method of the type of support desirable for the workability apparatus. Experimental studies show that variable results are obtained when the rammer is placed on table tops of various construction; consequently it should be securely mounted on a mass of substantial weight. This feature was originally pointed out by workers in the foundry sand field who use the apparatus. Accordingly, the committee recommends that the following paragraph be published as a tentative revision of this standard:

Section 2.—Add a new Paragraph (*c*) to read as follows:

> (*c*) The rammer shall be mounted on a concrete rectangular column measuring at least 8 by 11 in. and 27 in. in height. Four ¼-in. bolts, at least 3 in. in length, shall be cast in the top of the column for use in holding the rammer, and the space between its base and the top of the column shall be grouted with cement mortar. The apparatus may be placed on the floor and can then be conveniently operated from a sitting position. Variable results may be obtained from the test unless a suitable mounting is used for the rammer.

VI. STANDARDS CONTINUED WITHOUT REVISION

Standard Methods of Test for Cold Crushing Strength and Modulus of Rupture of Refractory Brick and Shapes **(C 133 - 39)**.[1]—Although this standard has stood for six years without revision, it is still in accord with present practice. The committee therefore recommends its continuation as standard.

The recommendations appearing in this report were submitted to letter ballot of the committee, which consists of 34 members, with the results shown in Table V.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Industrial Survey (L. C. Hewitt, chairman) is reviewing the open-hearth survey, recently revised by W. S. Debenham. The survey on refractories for coke ovens is undergoing revision by W. C. Reuckel, and that for the lead industry by F. E. Wormser of the Lead Industries Association. The survey of refractories in the copper industry will be rewritten as soon as the subcommittee can get someone to undertake the job.

A new survey on steel mill pouring pit refractories is being prepared by L. G. Ekholm and L. D. Hower, Jr., and the United Engineers and Constructors have agreed to assign a man to prepare a survey on linings for water-gas sets. Arrangements have not yet been made for the handling of proposed new surveys on refractories for incinerators and for the paper industry.

Subcommittee II on Research (R. B. Sosman, chairman) is planning to publi-

TABLE V.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. New Standard, Immediate Adoption			
Test for Permanent Linear Change Upon Reheating of Insulating Fire Brick	30	0	1
II. Revision of Standards, Immediate Adoption			
Methods of Testing Insulating Fire Brick (Compressive Strength, Flexural Strength, and Permanent Linear Change After Heating) (C 93 - 42)	30	0	1
Test for Permanent Linear Change After Reheating of Refractory Brick (C 113 - 36)	30	0	1
Test for Apparent Porosity, Water Absorption, Apparent Specific Gravity and Bulk Density of Burned Refractory Products (C 20 - 41)	25	0	0
III. Adoption of Tentatives as Standard			
Test for Sieve Analysis and Water Content of Refractory Materials (C 92 - 43 T)	30	0	1
Test for Combined Drying and Firing Shrinkage of Fireclay Plastic Refractories (C 179 - 43 T)	29	0	2
Symbols for Heat Transmission (C 108 - 44 T)	31	0	0
IV. Adoption as Standard of Tentative Revisions of Standards			
Test for Pyrometric Cone Equivalent (P.C.E.) of Refractory Materials (C 24 - 42).	30	0	1
Def. of Terms Relating to Refractories (C 71 - 45)	30	0	1
V. Tentative Revisions of Standards			
Panel Test for Resistance to Thermal and Structural Spalling of Refractory Brick (C 38 - 45)	31	0	0
Panel Test for Resistance to Thermal and Structural Spalling of High Heat Duty Fireclay Brick (C 107 - 45)	31	0	0
Panel Test for Resistance to Thermal and Structural Spalling of Super Duty Fireclay Brick (C 122 - 45)	31	0	0
Spec. for Refractories for Malleable Iron Furnaces with Removable Bungs and for Annealing Ovens (C 63 - 41)	29	0	2
Panel Test for Resistance to Thermal and Structural Spalling of Fire Clay Plastic Refractories (C 180 - 45)	31	0	0
Spec. for Refractories for Heavy Duty Stationary Boiler Service (C 64 - 41)	29	0	2
Spec. for Refractories for Incinerators (C 106 - 41)	29	0	2
Spec for Refractories for Moderate Duty Stationary Boiler Service (C 153 - 41)	29	0	2
Def. of Terms Relating to Refractories (C 71 - 45):			
Definition of Fire Clay	28	2	1
Definition of Air-Setting Mortar	29	0	2
V. Tentative Revision of Standards—*Continued*			
Test for True Specific Gravity of Burned Refractory Materials (C 135 - 40)	30	0	1
Spec. for Fireclay Plastic Refractories for Boiler and Incinerator Services (C 176 - 44)	29	0	2
Test for Workability Index of Fireclay Plastic Refractories (C 181 - 45)	30	0	1

cize a series of research problems, the solution of which would be of value to more than one of the tests now under the jurisdiction of Committee C-8. Suggested problems include: The degree of uniformity of raw materials, the applicability of spectrographic analysis, the effect of the time factor in the load test, methods for measuring the true temperature of refractories, and the effect of atmospheric composition on the test results.

Subcommittee III on Tests (S. M. Phelps, chairman) is composed of the chairmen of the several sections described below. The activities of the subcommittee are indicated in the foregoing recommendations concerning tentative and standard methods of test, and are further discussed under the individual sections. Mr. Phelps has served as chairman of the subcommittee since the death of Mr. Harvey.

Section A on Load (J. D. Sullivan, chairman) tabulated the results of the questionnaire which was sent to every laboratory in the United States known to have a load-test furnace. Nineteen reported as having furnaces. From the information given in the replies, the section is preparing load test schedules for silica, basic, and neutral brick. Consideration is also being given to the magnitude of the load to be used and to the methods for making temperature readings.

Section B on Spalling (R. E. Birch, chairman) prepared the new paragraphs which were approved by the committee for incorporation in the spalling procedures to describe the method for removing spalls from the test brick. The section is now further revising the Method of Panel Test for Resistance to Thermal and Structural Spalling of Refractory Brick (C 38 – 45) to include some of the material contained in the Recommended Practice for the Panel Spalling Test as issued by the Refractories Fellowship, Mellon Institute. The section is also considering possible editorial changes to make clear that 3-in. brick may be used as well as 2½-in. specimens in the spalling tests. Another matter under study by the section is the suggestion that the ignition loss of fireclay plastic refractories be obtained by placing dried and weighed molded test brick on top of the test panel, reweighing them on completion of the test, thus permitting calculation of the spalling loss.

Section C on Temperature (J. L. Carruthers, chairman) will investigate various types of furnaces to determine the extent of the temperature variation, and as a result of this study will recommend a type of furnace to be used in the P.C.E. test so that values of the same order can be obtained on each sample. The possibility of having cone 32½ standardized by the National Bureau of Standards will be investigated. The section is planning to prepare a manuscript on methods, apparatus, and technique of temperature control for inclusion in the C-8 Manual as a source of information for measuring and controlling heat in the A.S.T.M. procedures.

Section E on Analysis (L. J. Trostel, chairman) reviewed the Methods of Chemical Analysis of Refractories (C 18 – 45) to insure adequate description of all equipment. The section considered a suggestion that a decrease in the number of potassium permanganate solutions of varying normality used in Methods C 18 would result in simplification without loss of accuracy. It was noted that such a change would necessitate the use of an objectionably large amount of one of the standard titrating solutions in the analysis of chrome refractories, and the suggestion was rejected as applying to this one procedure.

Section F on Tests on Refractory Insulation (W. R. Kerr, chairman) prepared the proposed revision of Standard Methods of Testing Insulating Fire Brick (C 93 – 42) and the proposed new standard for permanent linear change, both of which were approved by the committee. The section will study suggestions for possible improvement of the linear change procedure through the use of small test specimens.

In considering the advisability of adding a group 30 to the present classification of insulating fire brick, the section decided against any recommendation at this time, because of the very limited number of brands on the market. The section also decided against adoption of a load test for evaluating insulating fire brick, as being of little value from the standpoint of behavior in service.

Section G on Porosity and Permanent Volume Change (C. E. Fulton, chairman) made the improvements in Methods C 113, C 20, and C 135, as described earlier in this report under the recommendation on standards.

The measurement of changes taking place in refractories during the "reheat test" has in recent years been made both on a volume and linear basis. The section is investigating the desirability of reporting volumes in Method C 113. This will involve obtaining a suitable means for measuring the volume of test brick and determining the order of accuracy of the results as compared with carefully made linear measurements.

Section II on Mortars and Plastic Refractories (R. S. Bradley, chairman) recommended the adoption as standard of Method C 179 – 43 T, and prepared the tentative revision of Method C 181 specifying a suitable mounting of the workability apparatus. Both of these actions were approved by Committee C-8.

Subcommittee IV on Heat Transfer (R. H. Heilman, chairman) is continuing to investigate methods for determining thermal conductivity. Mr. Heilman will contact the National Bureau of Standards to determine if the research program, which was interrupted by the war, can be resumed.

Subcommittee V on Precision and Tolerance (W. S. Debenham, chairman) prepared the paragraphs and tables which were approved by the committee for incorporation in the several C-8 specifications on the subject of the size and selection of the sample to be used in testing.

R. A. Heindl served as temporary chairman of this subcommittee until the appointment of W. S. Debenham at the C-8 meeting in November.

Subcommittee VI on Nomenclature (R. A. Heindl, chairman) made the recommendations in connection with the definitions in Standard C 71. The subcommittee is now preparing a definition for refractories comprised of chrome and magnesite, and also will develop definitions of special refractory terms in conjunction with the program of the new Subcommittee on Special Refractories.

Subcommittee VIII, Editorial (L. J. Trostel, chairman) has tentative plans for preparing a new edition of the Manual of A.S.T.M. Standards on Refractory Materials for publication in the spring of 1947. The subcommittee prepared various editorial changes in the standards and tentatives under the jurisdiction of Committee C-8.

Subcommittee IX on Classifications (S. M. Swain, chairman) has under study various suggestions made in regard to the classification of the several grades of fireclay refractories under the Classification of Fireclay Refractories (C 27 – 41).

Subcommittee X on Petrography (H. M. Kraner, chairman) is adapting, for inclusion in the next edition of the C-8 Manual, a manuscript on elementary procedures for microscopic examination of refractory materials.

Subcommittee XI on Special Refractories (M. C. Booze, chairman) is organizing a three-fold program involving the preparation of definitions, test procedures, and specifications. Definitions will be prepared by the Subcommittee on Nomenclature, while specifications will not be drawn up until the testing procedures are available. Test methods to be devised include hot strength, shrinkage, and spalling and other types of resistance. The subcommittee plans to confine the initial work to silicon carbide, bonded fused alumina, bonded mullite, molten cast mullite, zircon, and the purer grades of magnesia. Leading manufacturers will be consulted before deciding upon the particular type or types of each of these to be included in the initial tests. The subcommittee will also consult consumers and research organizations interested in special refractories.

This report has been submitted to letter ballot of the committee, which consists of 34 members; 25 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. D. SULLIVAN,
Chairman.

L. J. TROSTEL,
Vice-Chairman.

S. M. PHELPS,
Secretary.

REPORT OF COMMITTEE C-9[*] ON CONCRETE AND CONCRETE AGGREGATES

Committee C-9 on Concrete and Concrete Aggregates held one meeting during the year, in Pittsburgh, Pa., on February 27, 1946. This was the first meeting of the full committee since June, 1944. Interim meetings were held: One by Subcommittee XVIII on Ready Mixed Concrete on March 2, 1944, one by the Advisory Committee on June 28, 1945, and one by Subcommittee V on Editorial and Definitions on January 15, 1946. In addition, there were meetings of certain special working committees and considerable committee work carried out by correspondence.

On account of the times, this lessening of activity requires no explanation. As a result, the committee has little to report at this time. However, a number of activities were initiated at the February meeting which may be completed at the meeting of the committee to be held in conjunction with the Annual Meeting of the Society in June.

This report discusses problems current with the committee, outlines those few items on which work has been completed, but is devoted principally to matters which will be considered by the committee at its June meeting and which may subsequently be presented to the Society for appropriate action.

In 1945, the committee suffered the loss, through death, of one of its most valued members, Frederick C. Lang. Mr. Lang had been a member of the Society, as representative of the Minnesota Highway Department, since 1920. He joined Committee C-9 in 1928. He was active in the committee's work, serving on several of its subcommittees. Mr. Lang was long associated with the Minnesota Highway Department and the University of Minnesota, having been Engineer of Materials and Research for the Highway Department and Associate Professor or Professor of Highway Engineering for the University since 1918.

Sanford E. Thompson Award.—The Sanford E. Thompson Award for a paper of outstanding merit on concrete and concrete aggregates presented to the Society will be made this year to Prof. C. W. Muhlenbruch of the Carnegie Institute of Technology for his paper on "The Effect of Repeated Loading on the Bond Strength of Concrete," published in the 1945 *Proceedings*.[1] This is the fifth Sanford E. Thompson Award, previous ones having been made in 1944, 1941, 1940, and 1939.

Current and Future Work

The committee is presently giving consideration to a major reorganization in its subcommittee structure. Action along these lines was started in 1942 and a definite plan was proposed. However, due to war conditions, it was felt that no useful purpose would be served by putting it into effect, and was tabled. A reexamination of the problem is now in progress.

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[*] Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 824 (1945).

Briefly, it is felt that the work of the committee is now delegated to too few subcommittees with, in many cases, a membership too large for efficient functioning. Further, it is felt that the scope of work of some of these subcommittees is too broad. In general, the aim is to spread responsibility for committee projects and to make assignments more specific and of a more tangible nature.

The committee has discontinued its Subcommittee VI on Relations of Characteristics of Materials and Mixtures to Properties of Concrete. This was done at the recommendation of the chairman of the subcommittee, after thorough discussion and in recognition of the fact that the assigned scope was too broad and so intangible as to make it difficult for the subcommittee to function.

Two new subcommittees have been established which, undoubtedly, will be continued under any reorganization which may be perfected. One of these is to deal with admixtures for concrete used as additives to the batch. The other is to deal with the problem presented by aggregates which react chemically with cement. The importance of the first has been greatly emphasized by the advent of air-entrainment in concrete. "Chemically reactive" aggregates have presented problems which have received much independent study but the need for correlation of information available, and to be developed, is apparent.

The committee foresees opportunity for much useful work. In the specifications field the committee's work leaves many gaps unfilled. For example, aggregates are required to consist of "durable particles" but, even for the test methods developed by the committee, no conclusions have been reached as to acceptable specifications' requirements. The same thing may be said with respect to certain deleterious substances, resistance to abrasion, and other properties of aggregates. Test methods, even those most commonly used, need further study—in some cases, further refinement and, in other cases, modifications to make them of more practical use.

New test methods need development. For example, freezing-and-thawing tests are widely used in studies of durability of aggregates and of concrete, but no procedure for the conduct of such tests has been developed. What constitutes "workability" of concrete is a question as old as concrete technology but, while independent research has been done, no tangible steps have been taken to evaluate that characteristic. Among other problems which may be mentioned as requiring attention are: volume changes of concrete due to temperature and moisture, permeability of concrete to water and water vapors, elastic properties of concrete and methods for their measurements, resistance to abrasion of concrete, resistance of concrete to aggressive solutions, and methods for evaluating the grading of aggregates. These are illustrative; many others might be mentioned and will suggest themselves. A committee with the title "Concrete and Concrete Aggregates" has a field of endeavor ahead of it, the limitations of which cannot be readily visualized.

RECOMMENDATIONS AFFECTING STANDARDS

The committee has considered the Standard Definitions of Terms Relating to Concrete and Concrete Aggregates (C 125 - 44) and has made certain revisions. It recommends that the Standard Definitions be withdrawn and that the revisions in these former standard definitions be published as Tentative Definitions of Terms Relating to Concrete and Concrete Aggregates (C 125 - 46 T), as appended hereto.[2]

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[2] These revised definitions were accepted by the Society and appear in the 1946 Book of A.S.T.M. Standards, Part II.

These recommendations have been submitted to letter ballot of the committee, which consists of 75 voting members, with the results shown in Table I.

With respect to the definition for admixture which appears in the proposed revision of Definitions C 125, Committee C-9 directs that definition to the particular attention of Committee C-1 on Cement with the request that it give it consideration.

The committee recommends for the consideration of Committee E-8 on Nomenclature and Definitions the following revision in the Standard Definition of the Term Sand (C 58 - 28):

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE			
Def. of Terms Relating to Concrete and Concrete Aggregates (C 125 - 46 T)	53	2	4
II. WITHDRAWAL OF STANDARD			
Def. of Terms Relating to Concrete and Concrete Aggregates (C 125 - 44)	56	0	3

Sand.—Change from its present form: namely,

> *Sand.*—The fine granular material (usually less than $\frac{1}{4}$ in. in diameter) resulting from the natural disintegration of rock, or from the crushing of friable sandstone rocks.

to read as follows:

> *Sand.*—The fine granular material usually smaller than $\frac{1}{4}$ in. resulting from the erosion of rock by natural agencies or from the mechanical reduction of weakly cemented sandstone. The term is often used with a qualifying adjective to denote the product of mechanical crushing, as for example "stone sand" or "slag sand."

REVISIONS UNDER CONSIDERATION AFFECTING STANDARDS[3]

Standard Specifications for Ready-Mixed Concrete (**C 94 - 44**):

There is a likelihood that, as a result of a recent meeting of Subcommittee XVIII on Ready-Mixed Concrete, the committee will recommend the withdrawal of the present Standard C 94 - 44 and the adoption as tentative of a revised draft. While the revisions to be made are important they do not change the principles of the present specifications.

Standard Method of Test for Structural Strength of Fine Aggregate Using Constant Water-Cement-Ratio Mortar (**C 87 - 44**):

Title.—Change to read as follows: "Standard Method of Test for Measuring Mortar-Making Properties of Fine Aggregates."

Section 1.—Change from its present form: namely,

> 1. This method of test covers the procedure for determining the "structural strength" of sands to be used as fine aggregate in concrete by means of a compression test on a mortar of plastic consistency and gaged to a definite water-cement ratio.

to read as follows:

> 1. This method of test covers the procedure for measuring the mortar-making properties of fine aggregate for concrete by means of a compression test on specimens made from a mortar of plastic consistency and gaged to a definite water-cement ratio. Its principal use is intended for the determination of the effect of organic impurities revealed by the colorimetric test.

New Section.—Add the following as a new Section 2, renumbering the subsequent sections accordingly:

> 2. *Basis of Comparison.*—The fine aggregate under test shall be compared, in mortar as described in this test method, with graded standard sand having a fineness modulus of 2.40 ± 0.10. The graded sand shall consist of a mixture of approximately equal parts by weight of standard Ottawa sand conforming to the requirements prescribed in the Standard Method of Test for Tensile Strength of Hydraulic-Cement Mortars (A.S.T.M. Designation: C 190) and graded Ottawa sand conforming to the requirements

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[3] See Addendum to this report, p. 317.

specified in Section 4 of the Standard Method of Test for Compressive Strength of Portland Cement Mortars (A.S.T.M. Designation: C 109).

Section 3(a).—Revise the second sentence which now reads, "The flowtable with the attached shaft shall weigh 9 ± 1 lb.," by changing the weight to "9 ± 0.1 lb."

Standard Specifications for Concrete Aggregates (**C 33 - 44**):

Section 3(b).—Change from its present form: namely,

> (*b*) *Organic Impurities*.—All fine aggregate shall be free from injurious amounts of organic impurities. Aggregates subjected to the colorimetric test for organic impurities and producing a color darker than the standard shall be rejected unless they pass the mortar strength test as specified in Section 5.

to read as follows:

> (*b*) *Organic Impurities*.—All fine aggregate shall be free from injurious amounts of organic impurities. Aggregates subjected to the colorimetric test for organic impurities and producing a color darker than the standard shall be rejected unless, when tested in accordance with the Standard Method of Test for Measuring Mortar-Making Properties of Fine Aggregate (A.S.T.M. Designation: C 87), the mortar develops a compressive strength at 7 and 28 days of not less than . . . per cent[a] of that developed by the mortar specified in that method as the basis for comparison.

Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (**C 136 - 39**):

Section 3(a) and (b).—Change from their present form: namely,

> 3. (*a*) Samples for sieve analysis shall be obtained, by quartering or by use of a sampler, from a representative sample selected from the material to be tested.
>
> (*b*) Samples of fine aggregate for sieve analysis shall weigh, after drying, not less than the amount indicated in the following table:

Material with at least 95 per cent finer than a No. 10 (2000-micron) sieve....	100 g.
Material with at least 90 per cent finer than a No. 4 (4760-micron) sieve and more than 5 per cent coarser than a No. 10 (2000-micron) sieve..............	500 g.

to read as follows:

> 3. (*a*) Samples for sieve analysis shall be obtained from the materials to be tested by the use of a sample splitter or by the method of quartering. Fine aggregate sampled by the quartering method shall be thoroughly mixed and in a moist condition. The sample for test shall be approximately of the weight desired and shall be the end result of the sampling method. The selection of samples of an exact, predetermined weight shall not be attempted.
>
> (*b*) Samples of fine aggregate for sieve analysis shall weigh, after drying, approximately the amount indicated in the following table:

Material with at least 95 per cent finer than a No. 10 (2000-micron) sieve....	100 g.
Material with at least 90 per cent finer than a No. 4 (4760-micron) sieve and more than 5 per cent coarser than a No. 10 (2000-micron) sieve..............	500 g.

> In no case, however, shall the fraction retained on any sieve at the completion of the sieving operation weigh more than 4 g. per sq. in. of sieving surface.
>
> Note.—This amounts to 200 g. for the usual 8-in. diameter sieve. The amount of material retained on the critical sieve may be regulated by (*1*) the introduction of a sieve having larger openings than in the critical sieve, or (*2*) by the proper selection of the size of the sample.

Revisions under Consideration Affecting Tentatives[3]

Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (**C 88 - 44 T**):

Section 4(b).—Add a new sentence after the first sentence to read as follows: "Such sizes shall be tested in accordance with the procedure for fine aggregates."

Provide for two alternate groups of sizes of coarse aggretates. The first alternate would allow the use of suitably proportioned $\frac{1}{2}$- to 1-in. and 1- to 2-in. sizes in lieu of $\frac{3}{8}$- to $\frac{3}{4}$-in., $\frac{3}{4}$- to $1\frac{1}{2}$-in., and $1\frac{1}{2}$- to $2\frac{1}{2}$-in. sizes when the grading of the sample is such as to make them more appropriate. The second alternate would provide for the use of more closely graded sizes as follows:

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[a] A percentage should be inserted by the engineer to suit local conditions.

No. 4 to $\frac{3}{8}$ in.	300 g.
$\frac{3}{8}$ to $\frac{1}{2}$ in.	500 g.
$\frac{1}{2}$ to $\frac{3}{4}$ in.	750 g.
$\frac{3}{4}$ to 1 in.	1000 g.
1 to $1\frac{1}{2}$ in.	1500 g.
$1\frac{1}{2}$ to 2 in.	2000 g.
Larger sizes by 1-in. spread in sieve size, each fraction	3000 g.

This second alternate would be accompanied by a note reading as follows:

NOTE.—It should be noted that testing closely sized aggregates such as these constitutes a more severe test than testing a graded aggregate and this fact should be taken into account in establing limits in writing specifications.

Section 6(a).—Add the following note to this paragraph:

NOTE.—Suitably weighted wire grids placed over the sample in the containers will permit this coverage to be achieved with very lightweight aggregates."

Section 6(b).—Change the fourth sentence from its present form: namely, "The samples shall be dried to constant weight at the specified temperature." to read as follows: "The samples shall be dried for 5 to 7 hr. at the specified temperature."

Add a new Paragraph (*d*) to read as follows:

(*d*) *Length of Cycle.*—One cycle shall consist of an immersion period of 16 to 18 hr. followed by a drying period of 5 to 7 hr. and shall be completed within 24 hr. except that when the continuity of the cycle is interrupted the samples shall be stored at room temperature in a dry condition until the test is resumed.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, Kenneth B. Woods.
Vice-Chairman, Fred Hubbard.
Secretary, Stanton Walker.

This report has been submitted to letter ballot of the committee, which consists of 75 voting members; 59 members returned their ballots, of whom all have voted affirmatively.

Respectfully submitted on behalf of the committee,

F. E. RICHART,
Chairman.

STANTON WALKER,
Secretary.

ADDENDUM TO REPORT OF COMMITTEE C-9 ON CONCRETE AND CONCRETE AGGREGATES

The Report of Committee C-9 on Concrete and Concrete Aggregates, prepared prior to the annual meeting, refers to a number of revisions then under consideration. Actions were taken with respect to certain of them at the meeting of the committee held in Buffalo, N. Y., on June 27, 1946. These actions were presented at the general session of the Society on June 28, and adopted subject to letter ballot of the committee and of the membership of the Society. The items outlined in the original report on which affirmative actions were taken by the committee and by the Society are listed in Table II.

TABLE II.—ANALYSIS OF LETTER BALLOT VOTE

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. REVISION OF TENTATIVE			
Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 – 44 T):			
Section 4 (*b*) on Coarse Aggregate Samples	50	0	11
Addition of Grading Alternate A	47	1	13
Addition of Grading Alternate B	45	3	13
Revision of Section 6 (*a*)	50	0	11
Revision of Section 6 (*b*)[a]	31	16	14
Addition of Section 6 (*d*)[a]	31	16	14
II. ADOPTION OF TENTATIVE AS STANDARD			
Test for Abrasion of Gravel by Use of the Deval Machine, (D 289 – 42 T), as revised	55	0	6
III. REVISION OF STANDARD, IMMEDIATE ADOPTION			
Spec. for Concrete Aggregates (C 33 – 44)	54	0	4
Test for Structural Strength of Fine Aggregate Using Constant Water Cement Ratio Mortar (C 87 – 44)	55	0	3
Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 – 44).	48	0	10
Test for Sieve Analysis of Fine and Coarse Aggregates (C 136 – 39)	56	0	2

[a] These items failed to receive the necessary two-thirds affirmative vote for approval and accordingly were not adopted.

In addition, Committee C-9 concurred with Committee D-4 in revisions as given in the report of that Committee,[1] of two test methods under the joint jurisdiction of the two committees, namely, the Methods of Test for Abrasion of Coarse Aggregate by use of the Los Angeles Machine (C 131 – 44), and for Abrasion of Gravel by Use of the Deval Machine (D 289 – 42 T), Method D 289 to be adopted as standard.

These recommendations have been submitted to letter ballot of the committee, which consists of 72 voting members, with the results shown in Table II.

With respect to the Standard Specifications for Ready-Mixed Concrete (C 94 – 44), referred to in the original report, no final action was taken. The proposed revised specifications were referred back to Subcommittee XVIII with authority to present them, as revised, to the Society through the Administrative Committee on Standards.

This addendum has been submitted to letter ballot of the committee which consists of 72 voting members; 58 members returned their ballots of whom 55 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

K. B. WOODS,
Chairman.

STANTON WALKER,
Secretary.

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[1] See p. 367.

REPORT OF COMMITTEE C-12*

ON

MORTARS FOR UNIT MASONRY

The activities of Committee C-12 on Mortars for Unit Masonry were practically discontinued during the war and the committee held no meetings during 1945. However, a well-attended meeting was held in Washington, D. C., on March 7, 1946, during which the renewal of committee activities was discussed and a tentative program was outlined for various subcommittees.

The committee now consists of 34 members, of whom 13 are classified as producers, 7 as consumers and 14 as general interest members.

The present officers of the committee have been re-elected for the ensuing term of two years.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Research (F. O. Anderegg, chairman) plans research directed toward the development of reproducible tests of mortar, particularly determination of consistency and a "field test" of mortar.

Subcommittee III on Specifications and Methods of Tests for Mortar (H. C. Plummer, acting chairman) is now considering a specification for mortar proposed by the National Lime Association and expects to recommend a mortar specification to the committee for publication as tentative during the current year.

Working Subcommittee on Efflorescence (P. L. Rogers, chairman) has planned a series of tests to determine the efflorescing tendencies of mortars and hopes to establish correlation between the results of such tests and the performance of mortars in structures.

This report has been submitted to letter ballot of the committee, which consists of 35 members; 26 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

T. I. Coe,
Chairman.

H. C. Plummer,
Secretary.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

REPORT OF COMMITTEE C-14*
ON
GLASS AND GLASS PRODUCTS

Committee C-14 on Glass and Glass Products held one meeting during the year in Buffalo, N. Y., on May 1, 1946.

Subsequent to the 1945 Annual Meeting, Committee C-14 presented to the Society through the Administrative Committee on Standards a proposed revision of the Tentative Definition of The Term Glass (C 162 - 41 T) which was accepted[1] on June 27, 1945, the revised definition appears in the 1945 Supplement to Book of A.S.T.M. Standards, Part II, p. 147.

A special compilation of "A.S.T.M. Standards on Glass and Glass Products" will be published by the Society shortly after the Annual Meeting. This publication is sponsored by Committee C-14, and the Advisory Committee has carefully considered the material that should be included in the first edition of this publication.

The present officers of the committee and the members of the Advisory Committee have been re-elected for the ensuing term of two years.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Definitions (F. C. Flint, chairman).—This subcommittee prepared the revised Definition of the Term Glass (C 162 - 45 T) now published as tentative.

Subcommittee II on Chemical Analysis (G. E. F. Lundell, chairman).—The subcommittee is considering the preparation of standard methods for the chemical analysis of glass-making materials such as feldspar, fluorspar, and lime, as well as a standard sample of a suitable limestone for ignition, to yield lime as needed for such purposes. The National Bureau of Standards has offered standard analyzed samples of soda and potash, feldspars and fluorspars for use with these materials. The subcommittee will endeavor to recommend standard methods at the next Annual Meeting.

Subcommittee III on Chemical Properties (J. F. Greene, chairman).—The subcommittee is participating with Subcommittee VII in a comparative study of ten methods (four U.S. Pharmacopeia and six from Subcommittee III) on durability of glass and glass containers for the purpose of reducing the number of test methods to a minimum and to permit selection of a reasonable number of standard tests.

Subcommittee VI on Glass Construction Block and Tile (J. C. Kohl, chairman, succeeding J. P. Staples).—A new draft of Proposed Methods for Sampling and Testing Structural Non-Load-Bearing Cellular Glass Blocks has been prepared and submitted to the subcommittee preparatory to their submission to Committee C-14 for approval as tentative.

Subcommittee VII on Glass Containers (W. R. Lester, chairman).—Discussion is under way on the revision of the Standard Method of Hydrostatic Pressure Test on Glass Containers (C 147 - 43), Standard Method of Polariscopic Examination of Glass Containers (C 148 – 43), and Standard Method of Thermal Shock Test on Glass Containers (C 149 – 43), as a result of experience and knowledge gained through use over the past five years and from work in progress in several individual laboratories. Progress on the ring section examination method of glass containers is expected to be held up for at least two years due to lack of availability of suitable optical equipment. A suitable sampling method for glass containers is in progress, based on the experience of the Army Ordnance Department.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

[1] In submitting this recommendation to the Administrative Committee on Standards, Committee C-14 reported the following results of the letter ballot vote of a total of 39 ballots returned from a membership of 63: affirmative 31, negative 0, ballots marked "not voting" 8.

Subcommittee VII-A on Pharmaceutical Containers (F. C. Flint, chairman) has been working with the U.S.P. Committee in testing samples of several types of glass with the various standard methods of A.S.T.M. and U.S.P. Three of the six testing laboratories have already submitted results of their first test and, when results of the other three are in, statistical analyses of the data will be made and a meeting of the subcommittee will be held to determine the next series of tests. The hope is that the two methods can be made identical; thus the number of standards which the U.S.P. lists can be simplified.

Subcommittee VIII on Flat Glass (D. E. Sharp, chairman).—After a lapse of activity due to the war, projects have now been initiated toward the standardization of test methods and reports for light and heat transmission of heat-absorbing glass.

This report has been submitted to letter ballot of the committee, which consists of 51 members; 30 members returned their ballots, of whom 27 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

LOUIS NAVIAS,
Chairman.

S. R. SCHOLES,
Secretary.

REPORT OF COMMITTEE C-15*

ON

MANUFACTURED MASONRY UNITS

During the past year, Committee C-15 on Manufactured Masonry Units held one meeting on March 6, 1946, in Washington, D. C. The recommendations that were considered at this meeting appear in this report.

I. New Tentative

The committee recommends for publication as tentative new Specifications for Structural Clay Facing Tile, as appended hereto.[1]

TABLE I.—PERMISSIBLE VARIATIONS IN DIMENSIONS.

Standard Dimension, in.	Permissible Variations from Standard Dimension, max., plus or minus
Face Height or Length	
Up to 3	1/8
Over 3 to 5, incl.	3/16
Over 5 to 8, incl.	1/4
Over 8 to 12, incl.	5/16
Through the Wall Thickness	
Up to 4	1/8
Over 6 to 8, incl.	3/16
Over 8	1/4

Note.—For a list of modular sizes, see American Standard A62.3. All of the sizes listed in this standard are not produced in some parts of the United States and purchasers should ascertain the size or sizes available.

II. Tentative Revisions of Standards

Specifications for Building Brick (Made from Clay or Shale) (**C 62 – 44**):[2]

Title.—Place an asterisk in the title after "Building Brick" and add the following footnote: "*When the term brick is used in these specifications, it should be understood to mean brick or solid masonry units."

Section 3 (*a*).—Change to read as follows:

> 3. (*a*) *Size.*—The size of brick shall be as specified by the purchaser and the average size of brick furnished shall approximate the size specified in the invitation for bids. The maximum permissible variation in dimensions of individual units shall not exceed those given in Table II (the accompanying Table I).

Specifications for Sand-Lime Building Brick (**C 73 – 39**).[2]—The committee recommends the same revisions in these specifications as shown above for Specifications C 62 – 44.

TABLE II.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. New Tentative			
Spec. for Structual Clay Facing Tile[1]	36	0	6
II. Tentative Revisions of Standards			
Spec. for Building Brick (Made from Clay or Shale) (C 62 – 44)	31	0	11
Spec. for Sand-Lime Building Brick (C 73 – 39)	33	0	9
Methods of Sampling and Testing Brick (C 67 – 44)	31	0	11

Methods of Sampling and Testing Brick (**C 67 – 44**):[2]

Section 10 (*c*).—Change the last sentence from its present form "The caps shall be aged at least 2 hr. before the specimens are tested," to read "The caps should be aged at least 16 hr. before the specimens are tested."

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] See Editorial Note, p. 322.

[2] 1944 Book of A.S.T.M. Standards, Part II.

The recommendations appearing in this report have been submitted to letter ballot of the committee, which consists of 59 members, with the results shown in Table II.

ACTIVITIES OF SUBCOMMITTEES

All of the subcommittees of Committee C-15 have been somewhat inactive during the war but have indicated a great deal of interest in the various specifications under their jurisdiction, and it may be expected that the specifications will be closely scrutinized in the near future for any possible need of revision.

The present officers of the committee have been re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee, which consists of 59 members; 29 members returned their ballots, of whom 28 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

D. E. PARSONS,
Chairman.

J. W. WHITTEMORE,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C–15 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Revision of Standard Specifications for:
Building Brick (Made from Clay or Shale) (C 62 – 44),
Structural Clay Load-Bearing Wall Tile (C 34 – 41),
Structural Clay Non-Load-Bearing Tile (C 56 – 41), and
Structural Clay Floor Tile (C 57 – 39).

Tentative Revision of Standard Methods of:
Sampling and Testing Brick (C 67 – 44), and
Sampling and Testing Structural Clay Tile (C 112 – 36).

Revision of Standard and Reversion to Tentative:
Definitions of Terms Relating to Structural Clay Tile (C 43 – 46 T).

These recommendations were accepted by the Standards Committee on September 9, 1946, and appear in the 1946 Book of A.S.T.M. Standards, Part II.

On November 23, 1946, the Standards Committee accepted the Proposed Specifications for Structural Clay Facing Tile for publication as tentative. The new tentative specifications have been assigned the designation C 212 – 46 T and appear in the 1946 Book of A.S.T.M. Standards, Part II, p. 1321.

Committee C-15 presented to the Standards Committee new Tentative Specifications for Facing Brick (Solid Masonry Unit Made from Clay or Shale) which were accepted on September 9, 1946. On subsequent recommendation of Committee C-15 publication of these specifications as tentative has been withheld.

REPORT OF COMMITTEE C-16[*]

ON

THERMAL INSULATING MATERIALS

Committee C-16 on Thermal Insulating Materials and several of its subcommittees held meetings on only one occasion during the past year. This was during the Spring Group Meetings held the week of February 25, 1946, at Pittsburgh, Pa. At the meeting of the committee held on February 27, 1946, election of officers for the ensuing term of two years resulted in the selection of the following:

Honorary Chairman, J. H. Walker.
Chairman, Ray Thomas.
Vice-Chairman, B. A. Hollenbeck.
Secretary, K. M. Ritchie.
Advisory Committee, C. B. Bradley, P. D. Close, and R. H. Heilman.

Another important action taken at this meeting was the reinstatement of the program, in force before Pearl Harbor, of holding two meetings of subcommittees each year, which reinstatement is to be effective at least for 1946. It is hoped that the work of the various subcommittees will be expedited by this additional meeting.

I. NEW TENTATIVES

The committee recommends that the following specifications and test procedures be accepted for publication as tentative: Tentative Specifications for Structural Insulating Board Made from Vegetable Fibers,[1] and Tentative Methods of Testing Structural Insulating Board Made from Vegetable Fibers.[1]

II. TENTATIVES CONTINUED AS TENTATIVE

The committee recommends that the following specifications and methods that have remained in the tentative status for two years or less without revision be continued as tentative:

Tentative Specifications for:

85 per cent Magnesia Thermal Insulating Cement (C 193 - 44 T),
Asbestos Thermal Insulating Cement (C 194 - 44 T),
Mineral Wool Thermal Insulating Cement (C 195 - 45 T),
Expanded or Exfoliated Vermiculite Thermal Insulating Cement (C 196 - 44 T),
Diatomaceous Silica Thermal Insulating Cement (C 197 - 44 T), and

Tentative Method of Test for:

Flexural Strength of Preformed Block Type Thermal Insulation (C 203 - 45 T).

The recommendations appearing in this report have been submitted to letter ballot of the committee, which consists of 54 voting members; 46 members returned their ballots, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
NEW TENTATIVES			
Spec. for Structural Insulating Board Made from Vegetable Fibers	38	0	8
Methods of Testing Structural Insulating Board Made from Vegetable Fibers	37	3	6

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[*] Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

[1] These methods and specifications were accepted as tentative by the Society and appear in the 1946 Book of A.S.T.M. Standards, Part II.

ACTIVITIES OF SUBCOMMITTEES

There has been no change from previous reports in the scope of the work being undertaken by the various subcommittees. Changes in chairmanships have been made in some cases. Listed below are the subcommittees with their present chairmen.

Subcommittee I on Physical Properties of Preformed Block Insulation, Including Pipe Covering, C. B. Bradley, chairman.

Subcommittee II on Physical Properties of Structural Insulating Board made from Both Mineral and Vegetable Material, P. D. Close, chairman.

Subcommittee III on Physical Properties of Thermal Insulating Cement, H. W. Greider, chairman.

Subcommittee IV on Physical Properties of Blanket and Semirigid Insulation Made from Both Mineral and Vegetable Material, R. E. Cryor, chairman.

Subcommittee V on Physical Properties of Loose-Fill Insulating Materials Made from Both Mineral and Vegetable Material, J. F. White, chairman.

Subcommittee VI, A.S.T.M. Section of Joint Committee on Thermal Conductivity of All Forms of Insulation, F. B. Rowley, chairman.

Subcommittee VII on Measurement of Thermal Properties Other than Thermal Conductivity of Thermal Insulating Materials, G. M. Rapp, chairman.

Subcommittee VIII on Studies of Dimensional Standards of Preformed Insulation, Ray Thomas, chairman.

Subcommittee IX, A.S.T.M. Section of Joint Committee on Studies of Vapor Barriers Applied to Thermal Insulation Used Especially in the Air Conditioning and Refrigeration Fields, (A joint committee having representatives on it from A.S.T.M., A.S.R.E., A.S.H.V.E., and T.A.P.P.I.), R. H. Heilman, chairman.

Subcommittee X on Editorial Functions, B. A. Hollenbeck, chairman.

Subcommittee XI, Liaison with Committee C-8 on Refractories, R. H. Heilman, chairman.

All of these subcommittees have carried on their assigned work during the past year.

This report has been submitted to letter ballot of the committee, which consists of 54 members; 40 members returned their ballots, of whom 39 voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

E. T. COPE,
Chairman.

RAY THOMAS,
Secretary.

REPORT OF COMMITTEE C-18*

ON

NATURAL BUILDING STONES

Committee C-18 on Natural Building Stones has held no meetings since the presentation of its previous report[1] to the Society in 1942 because most of the members have been engaged in emergency work which prevented their participation in the less urgent matters with which this committee has been concerned. With the expected return of normal construction operations involving stone there will be renewed interest in the various matters before the committee.

Since the 1942 report was presented there have been five resignations including four producers and one consumer. The committee now consists of 16 members, of whom 4 are classified as producers, 6 as consumers, and 6 as general interest members.

Recently Subcommittee III on Test Procedures has taken steps to make certain revisions in the Standard Methods of Test for Absorption and Apparent Specific Gravity of Natural Building Stone (C 97 – 36) in order to bring them more in line with similar procedures on other materials. Other test methods which have stood for several years without change will also receive consideration.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, Oliver Bowles.
Vice-Chairman, T. I. Coe.
Secretary, D. W. Kessler.

ADOPTION OF TENTATIVE AS STANDARD

The committee recommends the adoption as standard of the Tentative Method of Test for Compressive Strength of Natural Building Stone (C 170 – 41 T)[2] and accordingly asks that this recommendation be approved for reference to letter ballot of the Society.

This recommendation was submitted in 1944 to letter ballot vote of the committee, which consisted of 16 members all of whom returned their ballots, 15 voting affirmatively, 0 negatively, and 1 member marked his ballot "not voting.'

This report has been submitted to letter ballot of the committee, which consists of 16 members; 16 members returned their ballots, of whom 14 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. C. CLARK,
Chairman.

D. W. KESSLER,
Secretary.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 285 (1942).

[2] 1944 Book of A.S.T.M. Standards, Part II.

REPORT OF COMMITTEE D-1*
ON
PAINT, VARNISH, LACQUER, AND RELATED PRODUCTS

Committee D-1 on Paint, Varnish, Lacquer, and Related Products held one meeting during the year on February 27, 1946, in Pittsburgh, Pa.

During the past year Subcommittee XIII on Shellac has again become active in order to undertake the sponsorship of the American Standards Association activities in the United Nations Standards Coordinating Committee on matters pertaining to the grading and testing of shellac. A new Subcommittee XI on Resins, under the chairmanship of J. J. Mattiello, has been formed to develop methods of test and specifications for resins.

Subsequent to the 1945 Annual Meeting, Committee D-1 presented to the Society through the Administrative Committee on Standards a number of recommendations which are listed in Table I, together with the results of the letter ballot vote in Committee D-1 and the date of acceptance by the Standards Committee. The new and revised tentatives have been given the designations indicated and together with the tentative revisions appear in the February, 1946, compilation of "A.S.T.M. Standards on Paint, Varnish, Lacquer, and Related Products" and in the 1945 Supplement to Book of A.S.T.M. Standards.

RECOMMENDATIONS AFFECTING STANDARDS

Fourteen subcommittees have submitted reports of progress, seven of which cover recommendations affecting standards as a result of which the committee is submitting six new tentatives, revisions of three tentatives, revision of one standard for immediate adoption, revision of one standard and reversion to tentative, and is recommending the adoption of five tentatives as standard

TABLE I.—RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS, AUGUST 27, 1945, INCLUDING RECORD OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
New Tentative Methods of:			
Test for Evaluating Degree of Resistance of Traffic Paints to Abrasion (D 821 – 45 T)	99	0	50
Producing Films of Uniform Thickness of Organic Finishing Materials on Test Panels (D 820 – 45 T)	98	0	51
New Tentative Recommended Practice for:			
Operating Light and Water Exposure Apparatus (Carbon-Arc-Type) for Testing Paint, Varnish, Lacquer, and Related Products (D 822 – 45 T)	96	0	53
Revised Tentative Methods of:			
Test for Phthalic Anhydride Content of Alkyd Resin Solutions (D 563 – 45 T)	92	0	57
Preparation of Steel Panels for Exposure Tests of Enamels for Exterior Service (D 609 – 45 T)	105	2	42
Tentative Revisions of Standard Specifications for:			
Pure Chrome Green (D 212 – 41)	96	0	53
Chrome Oxide Green (D 263 – 41)	96	0	53
Prussian Blue (D 261 – 41)	93	0	56
Ultramarine Blue (D 262 – 41)	87	0	62
Titanium Dioxide Pigments (D 476 – 41)	93	3	54
Bone Black (D 210 – 41)	86	0	63
Carbon Black (D 561 – 41)	88	0	61
Lampblack (D 209 – 41)	89	1	59
Tentative Revisions of Standard Methods of:			
Chemical Analysis of White Pigments (D 34 – 39)	93	0	56
Test for Consistency of Exterior House Paints and Enamel-Type Paints (D 562 – 44)	101	0	48

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

without revision and two with revision, and the adoption as standard of seven tentative revisions of standards. The committee is also submitting for publication a new tentative (formerly an emergency standard) with editorial revisions.

These recommendations have been submitted to letter ballot of the committee which consists of 265 members, with the results shown in Table II.

TABLE II.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVES			
Evaluating Degree of Resistance of Traffic Paint to Bleeding...	85	0	33
Evaluating Degree of Settling of Traffic Paints	86	1	38
Spec. for Pumice Pigment.......	71	0	54
Test for Cellulose Acetate......	75	1	48
Test for Changes in Protective Properties of Coatings of Paint, Varnish, Lacquer, and Related Products on Steel Surfaces When Subjected to Immersion (formerly issued as Emergency Method ES – 35)..	87	0	31
Spec. for Titanium Dioxide Pigments (D 476 – 46 T).........	86	2	34
II. REVISION OF TENTATIVES			
Test for Dry to No-Pick-Up Time of Traffic Paint (D 711 – 43 T)................	86	0	39
Spec. for Red and Brown Iron Oxide Pigments (D 84 – 43 T)	82	1	42
Spec. for Yellow Iron Oxide, Hydrated (D 768 – 44 T)......	81	1	42
III. REVISION OF STANDARD AND REVERSION TO TENTATIVE			
Chemical Analysis of Zinc Yellow Pigment (Zinc Chromate Yellow) (D 444 – 39).............	85	0	39
IV. TENTATIVE REVISIONS OF STANDARDS			
Sampling and Testing Lacquer Solvents and Diluents (D 268 – 44)................	82	0	44
Spec. and Tests for Soluble Nitrocellulose (D 301 – 33) ...	74	0	52
V. ADOPTION OF TENTATIVES AS STANDARD			
Spec. for Oiticica Oil (Permanently Liquid) (D 601 – 41 T), as revised	77	0	48
Test for Light Sensitivity of Traffic Paint (D 712 – 43 T)..	82	0	41
Conducting Road Service Tests on Traffic Paint (D 713 – 43 T)	79	0	45
Def. of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 – 44 T), as revised	105	2	16
Spec. for Methyl Ethyl Ketone (D 740 – 43 T)...........	86	0	38
Spec. for Isopropyl Alcohol (D 770 – 44 T)............	78	0	45
Test for Water in Lacquer Solvents and Diluents (D 268 – 44 T).....................	79	0	44
VI. REVISION OF STANDARD, IMMEDIATE ADOPTION			
Spec. for Tricresyl Phosphate (D 363 – 36)...............	74	0	52
VII. ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS			
Spec. for Lampblack (D 209 – 41)	88	1	37
Spec. for Bone Black (D 210 – 41)	86	1	37
Spec. for Carbon Black (D 561 – 41)................	88	1	37
Spec. for Prussian Blue (D 261 – 41)................	84	1	39
Spec. for Ultramarine Blue (D 262 – 41)............	83	1	40
Spec. for Pure Chrome Green (D 212 – 41)............	84	1	40
Spec. for Chrome Oxide Green (D 263 – 41)...............	85	1	40

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Drying Oils (Francis Scofield, chairman) recommends that the Tentative Specifications for Oiticica Oil (Permanently Liquid) (D 601 – 41 T)[1] be adopted as standard with a modification consisting of a reduction in the minimum requirement for viscosity from "18" to "13" poises.

The subcommittee is preparing specifications for dehydrated castor oil and is investigating methods for measuring the acetone insoluble or acetone number of heat-bodied linseed oils.

The subcommittee is studying the Standard Methods of Testing Drying Oils (D 555 – 41) with the view of bringing the methods up to date.

Subcommittee III on Bituminous Emulsions (E. M. Berger, chairman) is preparing methods of test for bituminous emulsions intended for use as protective coatings.

Subcommittee IV on Traffic Paint (G. W. Ashman, secretary) recommends the adoption as standard without revision of the Tentative Methods of Test for Light Sensitivity of Traffic Paint (D 712 – 43 T) and for Conducting Road Service Tests on Traffic Paint (D 713 – 43 T).

The subcommittee recommends for publication a new Tentative Method of Evaluating the Degree of Resistance of Traffic Paint to Bleeding[2] and a new Tentative Method for Evaluating Degree of Settling of Traffic Paint.[2]

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[1] 1944 Book of A.S.T.M. Standards, Part II.

[2] These methods and specifications were accepted as tentative by the Society and appear in the 1946 Book of A.S.T.M. Standards, Part II.

The subcommittee recommends the following revisions in the Tentative Method of Test for Dry to No-Pick-Up Time of Traffic Paint (D 711 - 43 T):[1]

Section 3 (b).—In the first sentence, after the word "film" add "without downward thrust by operator." In the third sentence, after "30 sec." add "(Note)." Add the following note to this section:

> NOTE.—Progressive softening of the rubber tire by solvent in the paint or solvent used to clean the wheel will affect the end point. No solvent shall be used to clean the wheel while tests are being run.

The subcommittee is continuing its work on the development of tests for abrasion, adhesion, flexibility and hardness of traffic paint.

The subcommittee is also continuing its study of methods of measuring the night visibility of traffic paints. In this connection the use of white lacquer-coated sandpaper of various degrees of coarseness is giving indication of being satisfactory for use as standards for the calibration of instruments and for use for the visual ratings of traffic paints.

Subcommittee V on Volatile Solvents for Organic Protective Coatings (M. B. Chittick, chairman) is developing a procedure for evaluating hydrocarbons as thinners for protective coatings. The subcommittee is considering proposing specifications for heavy mineral spirits.

Subcommittee VI on Definitions (C. H. Rose, chairman) recommends that the Tentative Definitions of Terms Relating to Paint, Varnish, Lacquer and Related Products (D 16 - 44 T)[1] be adopted as standard with the following revisions, and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these modifications may be referred to letter ballot of the Society:

Resin.—Change the items under this heading to read as follows:

Natural Resin.—A solid organic substance originating in the secretion of certain plants or insects which is thermoplastic, flammable, non-conductive of electricity; breaks with a conchoidal fracture (when hard) and dissolves in certain specific organic solvents but not water.

Fossil Resin.—A natural resin of ancient origin usually found in the earth.

Synthetic Resin.—A synthetic substance physically similar to natural resin.

Urea Resin.—A synthetic resin made from urea and an aldehyde.

Acrylic Resin.—A synthetic resin made from derivatives of acrylic acid.

Vinyl Resin.—A synthetic resin made from vinyl compounds.

Styrene Resin.—A synthetic resin made from vinyl benzene.

Melamine Resin.—A synthetic resin made from melamine and aldehyde.

Maleic Resin.—A resin made from a natural resin and maleic anhydride or maleic acid.

Ester Gum.—A resin made from rosin and glycerine.

Alkyd Resin.—A synthetic resin made from polyhydric alcohols and polybasic acids; generally modified with resins, fatty oils or fatty acids.

Phenolic Resin.—A synthetic resin made from phenols and aldehydes.

Lacquer.—Delete the definition of "lacquer" including "sanitary or tin plate lacquers" and add the following:

> *Cellulose Lacquer.*—A liquid coating composition containing as the basic film-forming ingredients cellulose esters or ethers and plasticizers with or without resins.

Subcommittee VII on Accelerated Weathering Tests for Protective Coatings (H. A. Nelson, chairman) recommends that the Emergency Method of Test for Changes in Protective Properties of Coatings of Paint, Varnish, Lacquer, and Related Products on Steel Surfaces When Subjected to Immersion (ES - 35)[1] be published as tentative with an editorial change consisting of replacing the procedure for preparing the panels with the procedure given in the Tentative Method of Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products (D 609 - 45 T).[1]

The subcommittee has been cooperating with the Cleveland Paint and Varnish Production Club on the study of accelerated testing methods and procedures.

The subcommittee is considering revisions in the Tentative Recommended Practice for Operating Light and Water Exposure Apparatus, Carbon-Arc Type (D 822 - 45 T) and the Tentative Method of Preparation of Steel Panels for Testing Paint, Varnish, Lacquer and Related Products (D 609 - 45 T) and, therefore, is making no recommendation for a change in status of these methods.[3]

The subcommittee is preparing a list of special requirements that are unique in the testing of paints by the salt-spray testing apparatus and this material will be recommended for inclusion as a supplement or as a revision of the Tentative Method of Salt Spray (Fog) Testing (B 117 - 44 T).

Subcommittee VIII on Methods of Chemical Analysis of Paint Materials (C. Y. Hopkins, chairman) recommends that the following 16 standards which have stood for six years without revision be continued as standard:

Methods of Test for:

Chemical Analysis of White Pigments (D 34 - 39),
Chemical Analysis of Yellow, Orange, Red, and Brown Pigments Containing Iron and Manganese (D 50 - 36),
Flash Point by Means of the Tag Closed Tester (D 56 - 36),
Chemical Analysis of Yellow and Orange Pigments Containing Chromium Compounds, Blue Pigments and Chrome Green (D 126 - 36),
Specific Gravity of Pigments (D 153 - 39),
Alkalinity or Acidity of Pigments (D 278 - 31),
Bleeding of Pigments (D 279 - 31),
Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments (D 280 - 33),
Oil Absorption of Pigments (D 281 - 31),
Chemical Analysis of Dry Cuprous Oxide (D 283 - 39),
Chemical Analysis of Dry Mercuric Oxide (D 284 - 33),
Acetone Extract in Dry Lampblack and Dry Bone Black (D 305 - 31),
Tinting Strength of White Pigments (D 332 - 36),
Relative Dry Hiding Power of Paints (D 344 - 39),
Mass Color and Tinting Strength of Color Pigments (D 387 - 36),
Relative Dry Hiding Power of White Pigments in a Linseed Oil Vehicle (D 406 - 39),

The subcommittee recommends that the Standard Methods of Chemical Analysis of Zinc Yellow Pigment (Zinc Chromate Yellow) (D 444 - 39)[1] be reverted to tentative with a revision consisting of deleting the first two sentences in Section 6 (*b*). The subcommittee is considering further revisions in this method.

Subcommittee IX on Varnish (W. T. Pearce, acting chairman) is continuing its evaluation of the several methods for measuring the drying of varnish and resin films and is also studying methods for the determination of polybasic acids other than phthalic in alkyd resin solutions.

The subcommittee has completed a survey of methods for testing coating resins and is considering revisions in the Standard Methods of Testing Oleoresinous Varnishes (D 154 - 43).

Subcommittee X on Optical Properties (M. Rea Paul, chairman) is developing methods of color specifications, methods of test for directional reflectance and transmittance of material, and also methods for the preparation of panels for gloss determination.

The subcommittee is also working on methods to measure color differences and daylight and infra-red reflectance.

A method covering the preparation of panels for the determination of apparent luminous reflectance of traffic paints for night viewing is under preparation.

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[3] See Editorial Note, p. 336.

The subcommittee recommends that the Tentative Method of Test for Specular Gloss of Paint Finishes (D 523 – 44 T) be continued as tentative as consideration is being given to modifications in this method.

Subcommittee XIII on Shellac (C. C. Hartman, chairman) recommends continuance of the following in their present status:

Standard Specifications for Dry Bleached Shellac (D 207 – 35),
Tentative Method of Test for Color of Orange Shellac (D 29 – 41 T), and
Tentative Specifications for Orange Shellac and Other Lacs (D 237 – 43 T).

This subcommittee is prepared to undertake such cooperative work and exchange of information pertinent to any desired study for the development of international methods for the sampling and grading of shellac which may be referred to it by the United Nations Standards Coordinating Committee through the American Standards Association.

Subcommittee XV on Specifications for Pigments, Dry and in Oil, When Marketed in that Form (C. L. Crockett, chairman) recommends for publication new Tentative Specifications for Pumice Pigment, as appended hereto.[2]

The subcommittee also recommends revisions in the following two Tentative Specifications:

Tentative Specifications for Red and Brown Iron Oxide Pigments (**D 84 43 T**):[1]

Section 2 (b).—Change to read as follows:

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the specified pigment with linseed oil (with or without a small amount of volatile thinner) together with (where necessary) small amounts of wetting and dispersing agents to a semi-paste or fluid type consistency. As received, it shall not be caked in the container and shall break up readily in linseed oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile petroleum spirits of any mixtures of these substances. The paste shall conform to the following requirements:

Nonvolatile matter in the vehicle, min., per cent of vehicle	80
Moisture by distillation,[a] max., per cent	2
Coarse particles and skins (total residue retained on a No. 325 (44 – micron) sieve), max., per cent of the dry pigment	2

[a] Moisture by distillation shall be determined in accordance with the Standard Method of Test for Water in Petroleum Products and Other Bituminous Material (A.S.T.M. Designation: D 95).

In addition, the colors in oil prepared from class I pigment shall conform to the following requirements:

Pigment, min., per cent	60
Consistency by the Stormer viscosimeter:[a]	
At shearing rate of 100 revolutions per 30 sec., min., g	700 (125 KU)
At shearing rate of 100 revolutions per 35 sec., max., g	1200 (145 KU)

[a] Determined in accordance with the Standard Method of Test for Consistency of Exterior House Paints and Enamel-Type Paints (A.S.T.M. Designation: D 562).

Tentative Specifications for Yellow Iron Oxide, Hydrated (**D 768 – 44 T**):[1]

Section 2.—Add a new Paragraph (*b*) to read as follows, relettering the present Paragraph (*b*) as (*c*):

(*b*) *Paste in Oil.*—The paste in oil shall be made by thoroughly grinding the specified pigment with linseed oil (with or without a small amount of volatile thinner) together with (where necessary) small amounts of wetting or dispersing agents to a semi-paste or fluid type consistency. As received, it shall not be caked in the container and shall break up readily in linseed oil to form a smooth paint of brushing consistency. It shall mix readily in all proportions, without curdling, with linseed oil, turpentine, or volatile petroleum spirits or any mixtures of these substances. The paste shall conform to the following requirements:

Pigment, min., per cent	55
Nonvolatile matter in the vehicle, min., per cent of vehicle	80
Moisture by distillation,[a] max., per cent	2
Coarse particles and skins (total residue retained on a No. 325 (44-micron) sieve), max., per cent of the dry pigment	1.0
Consistency by the Stormer viscosimeter:[b]	
At shearing rate of 100 revolutions per 30 sec., min., g	700 (125 KU

At shearing rate of 100 revolutions per 35 sec., max., g. .	1200 (145 KU)

[a] Moisture by distillation shall be determined according to the Standard Method of Test for Water in Petroleum Products and Other Bituminous Material (A.S.T.M. Designation: D 95).

[b] Determined in accordance with the Standard Method of Test for Consistency of Exterior House Paints and Enamel-Type Paints (A.S.T.M. Designation: D 562).

The subcommittee recommends that the following seven standards be revised by including in each respective standard the tentative revision[4] submitted in August, 1945:

Standard Specifications for:

Lampblack (D 209 – 41),
Bone Black (D 210 – 41),
Carbon Black (D 561 – 41),
Prussian Blue (D 261 – 41),
Ultramarine Blue (D 262 – 41),
Pure Chrome Green (D 212 – 41), and
Chromium Oxide Green (D 263 – 41).

The subcommittee recommends that the current tentative revision of the Standard Specifications for Titanium Dioxide Pigments (D 476 – 41) be withdrawn and that a new tentative revision in the form of complete tentative specifications be issued as appended hereto.[2]

The subcommittee is considering revisions of the Standard Specifications for Toxic Ingredients in Anti-Fouling Paints (Dry Cuprous Oxide, Dry Mercuric Oxide) (D 277 – 31) and the Standard Specifications for Pure Linseed Oil Putty for Glazing (D 317 – 33) and recommends that these specifications be continued as standard.

The subcommittee also recommends that the following specifications be continued as tentative:

Tentative Specifications for:

Raw Umber (D 763 – 44 T),
Burnt Umber (D 764 – 44 T),
Raw Sienna (D 765 – 44 T),
Burnt Sienna (D 766 – 44 T),
Venetian Red (D 767 – 44 T), and
Black Synthetic Iron Oxide (D 769 – 44 T).

Subcommittee XVIII on Physical Properties of Materials (R. H. Sawyer, chairman) is studying methods of measuring the adhesion of protective coatings and is also studying methods for measuring film thickness.

The subcommittee is studying the subject of measuring the film hardness of protective coatings.

Subcommittee XXV on Cellulosic Coatings (R. M. Carter, chairman) recommends that the following tentatives be adopted as standard and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society:

Tentative Specifications for:[1]

Methyl Ethyl Ketone (D 740 – 43 T),
Isopropyl Alcohol (D 770 – 44 T), and

Tentative Method of Test for:[1]

Water in Lacquer Solvents and Diluents (D 268 – 44 T).

The subcommittee recommends for immediate adoption the revision appended hereto[5] in the Standard Specification for Tricresyl Phosphate (D 363 – 36), and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting so that this revision can be referred to letter ballot of the Society for adoption.

The subcommittee recommends a tentative revision of the Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (D 268 – 44) consisting of adding a method of test for the determination of the ester value of tricresyl phosphate. The subcommittee also recommends the following editorial change in Standard Methods D 268: In Section 29 (*a*) in the formula for solids concentration, change "vol. of solvent − vol. of toluene" to read "vol. of solvent + vol. of toluene."

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[4] 1945 Supplement to Book of A.S.T.M. Standards, Part II.

[5] These revised specifications were accepted by the Society and appear in the 1946 Book of A.S.T.M. Standards Part II.

The subcommittee recommends the following tentative revision of the Standard Specifications and Tests for Soluble Nitrocellulose (D 301 – 33):

Sections 5 and 6.—Change to read as follows:

5. *Apparatus.*—Use the du Pont Nitrometer which is illustrated in Figs. 1 to 4. Figure 2(*b*) shows the universal measuring tube, although the nitrocellulose measuring tube shown in Fig. 2(*a*) is preferable for testing nitrocellulose. If desired, both measuring tubes may be included in the assembly, by replacing the three-way glass manifold with a four-way manifold.

Procedure

NOTE.—The cellulose acetate mask described in Section 7 (*d*) must be worn during the generation and measurement of the gas as a precaution in case of an explosion.

6. (*a*) Calibrate the measuring tube accurately in the usual manner, using mercury as the calibrating liquid.

(*b*) Standardize the apparatus using c.p. KNO_3 recrystallized twice from distilled water, ground to pass a No. 100 (149-micron) sieve and dried for 2 to 3 hr. at 135 to 150 C. Fill the compensating, measuring, and reaction tubes and their connections with mercury. Run 20 to 30 ml. of H_2SO_4 (A.C.S. analytical grade, 94.5 ± 0.5 per cent) into the reaction bulb through the cup at the top and admit about 210 ml. of air. Close the stopcocks, shake the bulb well, and allow to stand overnight. This desiccates the air which is then run into the compensating tube until the mercury is about on a level with the 12.50 per cent mark on the measuring tube, the two tubes being held at the same height. Then seal the compensating tube using a small, blowpipe flame. Place 0.9 to 1.0 g. of the KNO_3 in a weighing bottle and weigh the latter accurately. Then transfer the KNO_3 to the cup of the reaction bulb and weigh the weighing bottle to obtain the weight of sample used. Add 1.0 ml. of water and stir the mixture in the cup with a small glass stirring rod to liberate the entrained bubbles of air; work the undissolved crystals into the lower part of the cup keeping them below the surface of the solution. It is not necessary that the nitrate dissolve before drawing it into the reaction bulb. *Make sure the lower stopcock is open,* then admit the mixture to the bulb by a series of quick openings of the upper stopcock, in the meantime keeping the crystals below the surface of the liquid. In this way, all but a small amount of the nitrate may be run into the bulb. Rinse the cup with a second 1.0-ml. portion of water, then repeat with a third 1.0-ml. portion (3 ml. in all). This should be sufficient to dissolve all remaining particles of nitrate in the cup. Transfer 25 ml. of the H_2SO_4 (94.5± 0.5 per cent), divided in several portions, to the cup, and subsequently to the bulb by lowering the reservoir slightly and opening and closing the upper stopcock, care being taken that no air enters even the bore hole in the stopcock. There must always be a slight suction when introducing the sample, the wash water and the acid, but never enough to cause air to be sucked into the reaction bulb. The quantities of water and H_2SO_4 used should be constant. Then with the bottom stopcock still open, lower the reservoir bulb to give reduced pressure in the reaction bulb and gently shake the reaction bulb to start the decomposition. After the evolution of NO has become slow (it is extremely important that the bottom stopcock be left open until the major part of the decomposition has occurred; otherwise, sudden evolution of gas will burst the bulb, scattering acid and glass) lower the reservoir bulb until all but 25 ml. of the mercury in the reaction bulb is withdrawn, close the bottom stopcock, and shake the reaction bulb vigorously for 5 min. When the reaction is completed allow the gas to cool for 20 min. then transfer the gas to the measuring tube. By means of the leveling device make careful adjustment of the mercury levels so that the mercury in the measuring tube is at the 13.85 per cent mark (the theoretical percentage of nitrogen in KNO_3) if an exactly 1.0000-g. sample was used, or a proportional reading if less was used. Paste a strip of paper on the compensating tube at the level of the mercury and the standardization is completed. It is advisable to make several check determinations, preferably on different days, to insure accurate standardization. Determinations should check within plus or minus 0.01 per cent.

(*c*) Dry the soluble nitrocellulose as for the ash determination described in Section 4 and place a sample of 1.0 to 1.2 g. in a weighing bottle. Dry at 100 to 105 C., stopper, cool in a desiccator, and weigh accurately. When the sample has been dried to constant weight (1 to 1½ hr. usually suffices), transfer it to the cup of the decomposition bulb, then reweigh the empty bottle to get the weight of the sample by difference. Add 5 to 10 ml. of H_2SO_4 (94.5 ± 0.5 per cent) to the cup and stir the mixture with a small stirring rod. Lower the mercury reservoir and then, *after making certain that the lower stopcock is open,* draw the mixture in by opening the upper stopcock. Take care that no air is drawn in. Rinse the cup of the decomposing bulb several times with H_2SO_4, using a total of 25 ml. for dissolving and rinsing. Complete the determination in accordance with the procedure described in Paragraph (*b*) for standardization of the apparatus, and take a reading after adjusting the level of the mercury in the reading tube to the mark on the compensating tube. The reading divided by the weight of sample gives the percentage nitrogen.

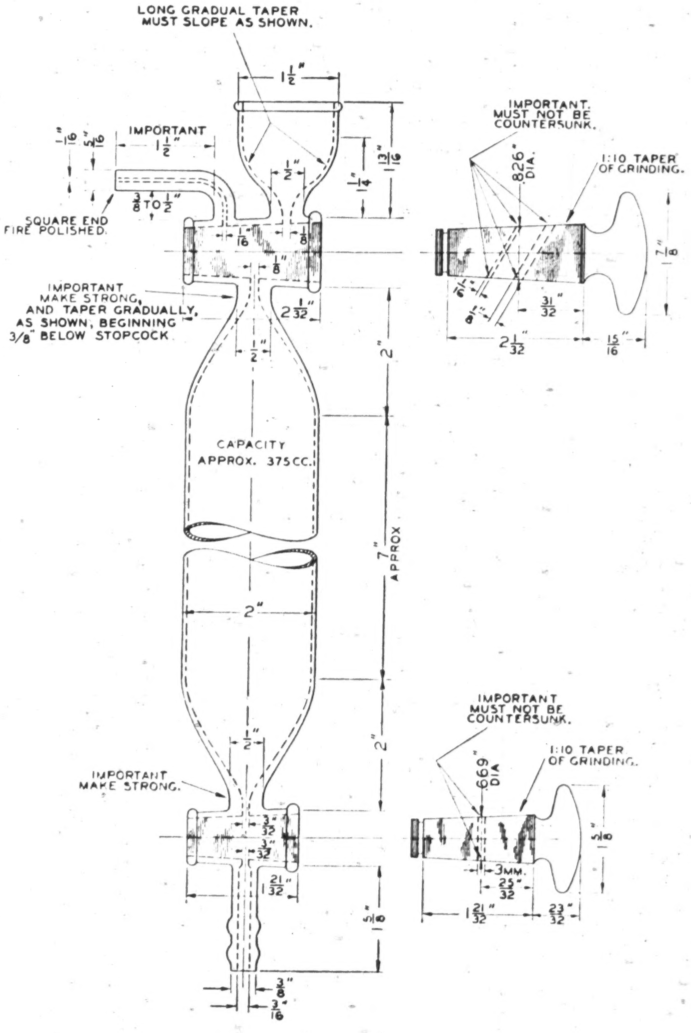

LONG GRADUAL TAPER MUST SLOPE AS SHOWN.
1 1/2"
IMPORTANT
1 1/2"
1/16"
5/16"
3/8 TO 1/2"
SQUARE END FIRE POLISHED.
1/2"
1 1/4"
1 13/16"
1/16
1/8
1/8"
IMPORTANT MAKE STRONG, AND TAPER GRADUALLY, AS SHOWN, BEGINNING 3/8" BELOW STOPCOCK
2 1/32"
1/2"
2"
CAPACITY APPROX. 375CC.
7" APPROX
2"
2"
1/2"
IMPORTANT MAKE STRONG.
3/32"
3/32"
1 21/32"
1 5/8"
3/8"
3/16
IMPORTANT. MUST NOT BE COUNTERSUNK.
.826" DIA.
1:10 TAPER OF GRINDING.
7/8"
1/16
1/8
31/32"
2 1/32"
15/16"
IMPORTANT MUST NOT BE COUNTERSUNK.
.669" DIA
1:10 TAPER OF GRINDING.
5/8"
3MM.
25/32"
1 21/32"
23/32"

FIG. 1.—Reaction Bulb.

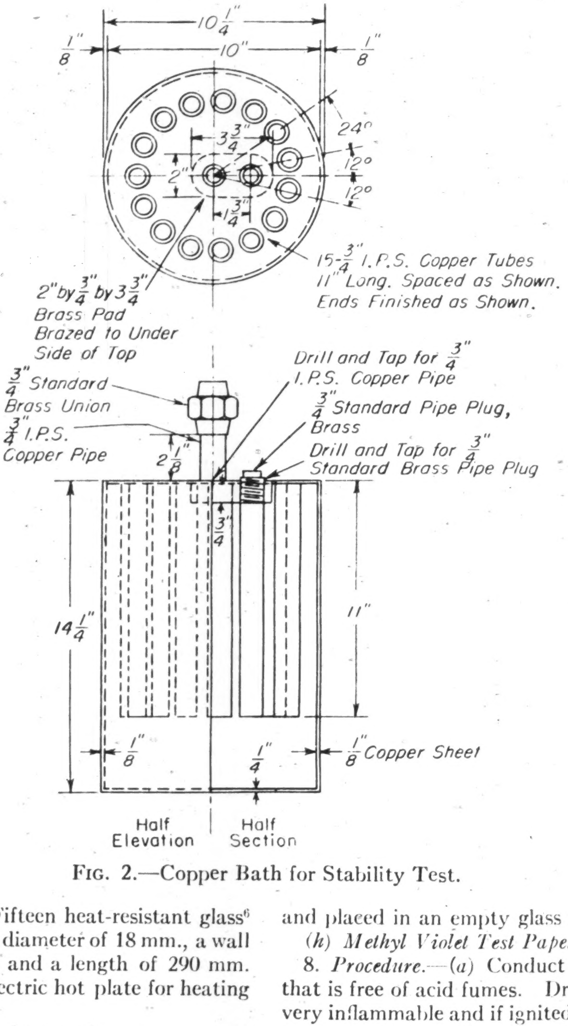
Fig. 3.—Replace the present Fig. 3 with the accompanying Fig. 1.

Sections 7, 8, and 9.—Change to read as follows:

7. *Apparatus.*—The apparatus shall consist of the following:

(*a*) *Copper bath.*—Copper bath with copper or brass condenser, as shown in Figs. 5 and 6. These baths are usually made to hold 13 to 15 test tubes. To aid in heat transfer, add 15 to 25 ml. of mineral oil to each copper well, in order to fill the space between the glass tube and the well. To maintain the bath at a temperature of 134.5

± 0.5 C., fill to within 3 in. of the top with a mixture consisting of 10 parts of a commercial ethylene glycol solution (automobile radiator antifreeze containing a corrosion inhibitor) and one part of water. Adjust by adding more glycol or water until the desired temperature is reached.



$10\frac{1}{4}''$
$\frac{1}{8}''$
$10''$
$\frac{1}{8}''$
$24°$
$3\frac{3}{4}''$
$12°$
$2''$
$12°$
$1\frac{3}{4}''$
15-$\frac{3}{4}''$ I.P.S. Copper Tubes $11''$ Long. Spaced as Shown. Ends Finished as Shown.
$2''$ by $\frac{3}{4}''$ by $3\frac{3}{4}''$ Brass Pad Brazed to Under Side of Top
Drill and Tap for $\frac{3}{4}''$ I.P.S. Copper Pipe
$\frac{3}{4}''$ Standard Brass Union
$\frac{3}{4}''$ Standard Pipe Plug, Brass
$\frac{3}{4}''$ I.P.S. Copper Pipe
$2\frac{1}{8}''$
Drill and Tap for $\frac{3}{4}''$ Standard Brass Pipe Plug
$\frac{3}{4}''$
$14\frac{1}{4}''$
$11''$
$\frac{1}{8}''$
$\frac{1}{4}''$
$\frac{1}{8}''$ Copper Sheet
Half Elevation
Half Section

FIG. 2.—Copper Bath for Stability Test.

(*b*) *Test Tubes.*—Fifteen heat-resistant glass[6] tubes, with an outside diameter of 18 mm., a wall thickness of 1.5 mm., and a length of 290 mm.

(*c*) *Heater.*—An electric hot plate for heating the bath.

(*d*) *Face Mask.*—A face mask, so constructed that a heavy piece of clear cellulose acetate sheeting protects the face.

(*e*) *Gloves.*—A pair of heavy gloves.

(*f*) *Pincers.*—Long pincers for handling the test tubes.

(*g*) *Thermometer.*—An A.S.T.M. Stability Test Thermometer having a range of 130 to 140 C. and conforming to the requirements for thermometer 26C - 42 as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1). The thermometer should be fitted with a cork stopper and placed in an empty glass tube in the bath

(*h*) *Methyl Violet Test Paper.*[7]

8. *Procedure.*—(*a*) Conduct the test in a room that is free of acid fumes. Dry nitrocellulose is very inflammable and if ignited by fire, spark, or static electricity will flare up and shatter the test tube. It is important, therefore, that the operator wear the cellulose acetate mask and heavy gloves and that the tubes be handled with long pincers.

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[6] **Pyrex glass** is satisfactory for this purpose

[7] The normal methyl violet test papers should be secured from the Naval Powder Factory, Indian Head, Maryland.

(*b*) Dry the sample overnight at room temperature followed by 30 min. at 40 to 45 C., or dry the wet material at 40 to 45 C. for a period of 4 to 5 hr. Weigh duplicate samples of 2.5 ± 0.1 g. into test tubes and press the samples down so that they occupy the lower 2 in. of the tubes, then swab out all nitrocellulose particles adhering to the inside wall of the tubes. Crease a piece (20 by 70 mm.) of normal methyl violet test paper for one-half its length, then insert in the tube with the uncreased portion downward, until the lower edge is 1 in. above the top of the sample. The paper must remain in this position throughout the test. Stopper the tube with a cork provided with a hole or notch 4 mm. in diameter. Place the tube, without jarring, in the heating bath maintained at 134.5 ± 0.5 C. Beginning at the end of the first 20 min., inspect the tube at 5-min. intervals by lifting the tube until the methyl violet paper, but not the nitrocellulose, is visible above the surface of the bath. The endpoint is reached when the entire test paper changes in color to salmon pink. For example, if the color is not completely changed at 20 min. but is completely changed at 25 min., record the stability of the sample as 25 min.

Figure 5.—Replace the present Fig. 5 with the accompanying Fig. 2.

Table I.—Delete the reference to "No. 5" in the requirements for 188–190 proof denatured alcohol.

The subcommittee also recommends for publication the new Tentative Methods of Test for Cellulose Acetate.[2]

The subcommittee recommends that the following ten standards which have stood for six years without revision be continued as standard:

Standard Specifications for:

Ethyl Acetate (85 to 88 per cent Grade) (D 302 – 33)
Amyl Acetate (Synthetic) (85 to 88 per cent Grade) (D 318 – 39),
Acetone (D 329 – 33),
Ethylene Glycol Monobutyl Ether (D 330 – 35),
Ethylene Glycol Monoethyl Ether (D 331 – 35),
Acetate Ester of Ethylene Glycol Monoethyl Ether (95 to 96 per cent Grade) (D 343 – 35),
Industrial 90 Benzene for Use in Paint, Varnish, Lacquer, and Related Products (D 361 – 36),
Industrial Grade Toluene for Use in Paint, Varnish, Lacquer, and Related Products (D 362 – 36),
Industrial Grade Xylene or Solvent Naphtha for Use in Paint, Varnish, Lacquer, and Related Products (D 364 – 36), and
Amyl Acetate Made from Fusel Oil (85 to 88 per cent Grade) (D 554 – 39).

Subcommittee XXIX on Painting of Structural Iron and Steel (A. J. Eickhoff, chairman) is considering initiating some new work in connection with the painting of structural metal.

Subcommittee on Membership (J. F. Broeker, chairman) is endeavoring to interest producers and consumers of paint products in the activities of Committee D-1 and to encourage members of Committee D-1 to increase their activities in subcommittee affairs.

Subcommittee on Papers (C. H. Rose, chairman).—At the conclusion of the Committee D-1 meeting on February 27, 1946, Mr. Peter Kass, of the Interchemical Corporation Research Laboratory, New York, N. Y. presented a paper "Analysis of Drying Oils by Ultra-Violet Spectrophotometry." The presentation of this paper had been arranged by the Papers Committee.

The present officers of Committee D-1 have been re-elected for the ensuing term of two years. In addition, J. C. Moore, C. L. Crockett, and G. G. Sward were elected members of the Advisory Committee.

This report has been submitted to letter ballot of the committee, which consists of 265 voting members; 134 members returned their ballots, of whom 93 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. T. PEARCE,
Chairman.

C. H. ROSE,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Revision of Standard Specifications for:

C.P. Zinc Yellow (Zinc Chromate) (D 478 – 41).

Revision of Tentative:

Method of Preparation of Steel Panels for Testing Paint ,Varnish, Lacquer, and Related Products (D 609 – 46 T),
Method of Test for Evaluating Degree of Settling of Traffic Paint (D 869 – 46 T), and
Recommended Practice for Operating Light and Water Exposure Apparatus (Carbon-Arc-Type) for Testing Paint, Varnish, Lacquer, and Related Products (D 822 – 46 T).

These recommendations were accepted by the Standards Committee on September 9, 1946, and the tentative revision and revised tentatives appear in the 1946 Book of A.S.T.M. Standards, Part II.

At the same time, the Standards Committee accepted the recommendation of the committee that the Emergency Method of Test for Changes in Protective Properties of Organic Coatings on Steel Surfaces when Subjected to Immersion (ES-35) be revised and issued as tentative. This new tentative appears in the 1946 Book of A.S.T.M. Standards, Part II, bearing the designation D 870 – 46 T).

REPORT OF COMMITTEE D-2*

ON

PETROLEUM PRODUCTS AND LUBRICANTS

Committee D-2 on Petroleum Products and Lubricants held two regular meetings during the past year (in January and in March, 1946, in Cleveland, Ohio).

Changes in Committee Organization.—During the year, subcommittee and technical committee changes were effectuated or authorized as follows:

Subcommittee XXII on Natural Gasoline (R. C. Alden, chairman) was discharged and its work was turned over to a new Technical Committee H on Light Hydrocarbons.

Subcommittee XXVIII on Autogenous Ignition Temperatures (A. H. Nuckolls, chairman) was discharged.

Technical Committee D on Stoddard Solvent (L. E. Jackson, chairman, and I. J. Fairchild, secretary) was discharged.

Technical Committee F on Diesel Fuel Oils (C. G. A. Rosen, chairman, and A. E. Becker, secretary) was suspended during reorganization.

Technical Committe H on Light Hydrocarbons (R. C. Alden, chairman) is still in course of organization.

A new Technical Committee I on Tractor Fuels has been authorized and is in course of organization.

A new Technical Committee J on Aviation Fuels has been authorized and is in course of organization.

A new Technical Committee K on Cutting Oils has been authorized and is in course of organization.

A new Coordinating Committee on Recommended Practices (H. C. Mougey, chairman) has been authorized and has held its organization meeting.

A Coordinating Committee on Significance of Tests (F. D. Tuemmler, chairman) has been authorized and will be organized to give continuous consideration to the revision of the publication, "Significance of Tests of Petroleum Products."

The membership of the Advisory Committee has been increased from nine to thirteen members.

Changes in Status of American Standards.—Table I shows the changes in status during the year of American Standards for petroleum products, approved by the American Standards Association on the recommendation of Sectional Committee Z11 on Petroleum Products and Lubricants.

ACTIVITIES OF TECHNICAL COMMITTEES AND SUBCOMMITTEES

Subcommittee X on Insoluble Content of Used Oils (L. L. Davis, chairman) has developed a Method of Test for Normal Pentane and Benzene Insolubles in Used Lubricating Oils. This subcommittee has unanimously recommended that this method, which is a revision of Emergency Method ES - 42, be published as tentative this year. This recommendation has been accepted, subject to letter ballot of Committee D-2, and if approved will be submitted to the Administrative Committee on Standards.[1]

Subcommittee XI on Determination of Inorganic Elements in Lubricants (C. M. Gambrill, chairman) found that the emergency alternate provisions (EA-D 129) in Standard Method of Test for Sulfur in Petroleum Oils by Bomb Method (D 129 – 44) are inadequate for many types of sulfur additives and has recommended that the emergency alternate provisions be discontinued. Because of the need for information on a method satisfactory for the determination of sulfur in oils containing additives, the subcommittee has developed a Method of Test for Sulfur in Lubricating Oils and in Additive Concentrates by Bomb Method. The method was found to require revision and was referred back to Subcommittee XI at the June meeting. It was decided to submit the revised method to letter ballot of Subcommittees VII and XI simultaneously, before submitting it to letter ballot of Committee D-2. Subject to favorable letter ballot of Committee D-2 the method will be referred to the Standards Committee for publication as tentative this year.[1] This method is being used currently with satisfactory results.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

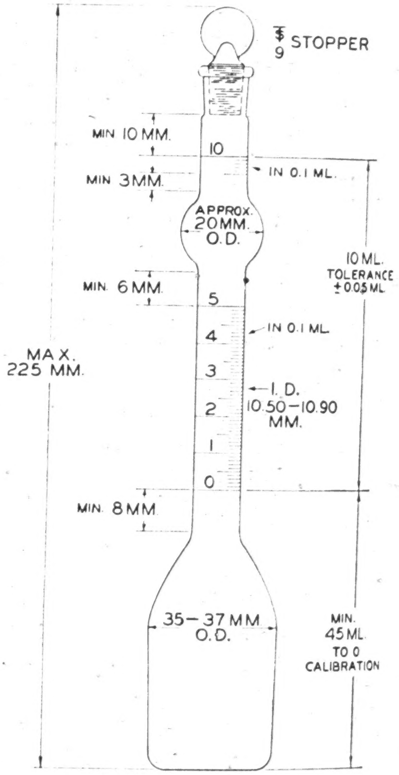
[1] **See Editorial Note, p. 348.**

TABLE I.

Title	A.S.T.M. Designation	ASA Number
REVISION OF AMERICAN STANDARDS		
Test for Carbonizable Substances in Paraffin Wax	D 612 – 45	Z11.50-1945
Test for Carbonizable Substances in White Mineral Oil (Liquid Petrolatum)	D 565 – 45	Z11.49-1945
Test for Distillation of Gasoline, Naphtha, Kerosine and Similar Petroleum Products......	D 86 – 45	Z11.10-1945
Test for Flash and Fire Points by Means of Open Cup	D 92 – 45	Z11. 6-1945
Test for Saponification Number of Petroleum Products by Color-Indicator Titration	D 94 – 45	Z11.20-1945
Test for Knock Characteristics of Motor Fuels	D 357 – 45	Z11.37-1945

Subcommittee XI has also submitted for publication as information the Proposed Method of Test for Sulfated Residue from Lubricating Oils (Indirect Acid-Oxidation Method) which appears in Appendix I.

The subcommittee found that the Tentative Method of Chemical Analysis for Phosphorus in Lubricating Oils (D 809 – 44 T) is inadequate for the determination of phosphorus in additives or in oils containing phosphorus in concentrations greater than 0.5 per cent. Pending the completion of tests on a more general method, the subcommittee has submitted for publication as information, the Proposed Method of Test for Phosphorus in Organic Materials (Acid Oxidation Method) which appears in Appendix II. This method gives promise of satisfactory results over the entire range of concentrations.

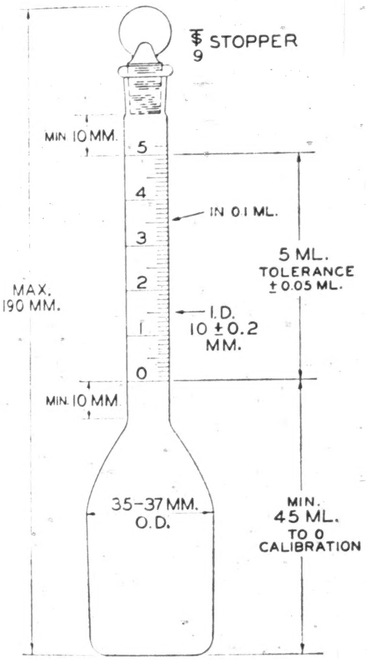


NOTE.—Alternate glass stopper similar to stopper shown in Fig. 1 may be used. Total height shall then be 190 mm. max.

FIG. 1.—Precision Sulfonation Flask, Range 0 to 50 Per Cent Absorption.

Subcommittee XXV on Analysis of Petroleum Products for Hydrocarbon Types (S. S. Kurtz, Jr., chairman) has developed the Proposed Method of Test for Aromatic Hydrocarbons in Mixtures with Naphthenes and Paraffins by Silica Gel Adsorption which appears as information in Appendix III.

This subcommittee recommends that the requirements for certain of the apparatus in the Proposed Method for Total Olefinic and Aromatic Hydrocarbons in Gasoline, as published for information in an appendix to the 1945 Report of Committee D-2[2], be revised as follows:



NOTE.—Alternate glass stopper similar to stopper shown in Fig. 1 may be used. Total height shall then be 190 mm. max.

FIG. 2.—Precision Sulfonation Flask, Range 48 to 100 Per Cent Absorption.

Fig. 1.—Change the statement underneath the drawing of the flask to read: "The volume of the flask to the zero calibration mark on the neck should be not less than 45 ml."

Fig. 2.—Replace this figure by the accompanying Fig. 1.

Fig. 3.—Replace this figure by the accompanying Fig. 2.

Section 3 (b).—Change tolerance on the 5-ml. pipette to read "± 0.02 ml." in place of "± 0.05 ml." For the 10-ml. pipette change the tolerance to read "± 0.04 ml." in place of "± 0.05 ml."

Note 3.—Change to read: "The standard two-cup, hand-driven, milk-test centrifuge for 6-in. Babcock bottles may be used when the standard sulfonation flasks (Fig. 1) are being centrifuged. The penny heads of the stoppers must be aligned in the same vertical plane to prevent breakage."

Technical Committee B on Lubricating Oils (W. S. James, chairman, and J. C. Geniesse, secretary) is sponsoring the Symposium on Oil Procurement Practices which is being held at the Annual Meeting of the Society in Buffalo, N. Y., during the week of June 24, 1946.

Technical Committee B has recommended for publication a new Tentative Method of Test for Foaming Characteristics of Viscous Petroleum Oils. The method was found to require revision and at the June meeting of Committee D-2, it was referred back to the technical committee. After letter ballot approval of the revised method by Technical Committee B it will again be submitted to Committee D-2 and then referred to the Standards Committee.[1]

Technical Committee C on Turbine Oils (F. C. Linn, chairman) has under development a Proposed Tentative Method of Test for Oxidation Characteristics of Steam Turbine Oils.

Technical Committee G on Lubricating Grease (F. L. Wright, chairman, C. W. Georgi, vice-chairman, and H. A. Mc-

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[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 247 (1945).

Conville, secretary) is sponsoring the exhibit of grease testing apparatus which is being displayed at the 1946 Annual Meeting of the Society.

I. NEW TENTATIVE METHODS

The committee recommends that the following three methods be accepted for publication as tentative:

Methods of:

Test for Sulfated Residue from New Lubricating Oils, prepared by Subcommittee XI on Determination of Inorganic Elements in Lubricants (C. M. Gambrill, chairman), as appended hereto.[3] This method represents a revision of and replaces Emergency Method ES - 43a.

Test for Olefins and Aromatics in Petroleum Distillates, prepared by Subcommittee XXV on Analysis of Petroleum Products for Hydrocarbon Types (S. S. Kurtz, Jr., chairman), as appended hereto.[3] This method represents a revision of and replaces Emergency Method ES - 45a.

Test for Oxidation Stability of Aviation Gasoline (Potential Gum Method), prepared by Technical Committee A on Gasoline (C. B. Veal, chairman), as appended hereto.[3]

II. REVISION OF TENTATIVE METHODS

The committee recommends revision of six tentative methods as follows:

Tentative Method of Test for Sulfur in Petroleum Oils by Lamp Method (**D 90 - 41 T**).[4]—Subcommittee VII on Sulfur Determination (H. M. Hancock, chairman) recommends the following revisions:

Section 2 (c).—Change to read as follows:

(*c*) *Absorber.*—An absorber of chemical-resistant glass conforming to the dimensions shown in Fig. 1, provided with standard-taper glass joints for connection with the chimney and spray trap. A fritted disk shall be sealed in the larger of the two bulbs of the absorber. The porosity of the fritted disk should be such that when 50 ml. of water are placed in the absorber and air is passed through the disk in an upward direction at a rate of 3.0 l. per min., the pressure differential between the two sides of the absorber is between 15 and 23 cm. of water, and the air is dispersed uniformly.

Fig. 1.—Change "Sintered Glass Plate (Average Pore Diameter 150–200 Microns)" to read "Fritted Disk."

To bring the dimensions into uniformity, the following changes are recommended:

	Change From	To Read
Section 2(*f*), line 10....	12 in.	30 cm.
Section 2(*g*), line 4....	8 in.	20 cm.
Section 2(*g*), line 5....	½ in.	13 mm.
Section 2(*g*), line 6....	¾ in.	19 mm.
Section 4(*d*), line 12....	½ in.	13 mm.
Section 4(*d*), line 12....	¾ in.	19 mm.
Fig. 2..................	12 in.	30 cm.
Fig. 3..................	8 in.	20 cm.
	2½ by 18 in.	65 by 450 mm.

Tentative Method of Test for Kinematic Viscosity (**D 445 - 42 T**).[4]—Subcommittee V on Viscosity (J. C. Geniesse, chairman) recommends the following changes:

Section 2.—Change the first sentence to read as follows: "The apparatus shall be of the capillary type and shall be capable, under proper manipulation, of measuring viscosity with an error not greater than specified in Section 5."

In the ninth line insert a footnote reference number "3" after "others," and add the following footnote, renumbering the subsequent footnotes accordingly:

[3] Other widely used viscosimeters are described in the following articles:

E. L. Ruh, R. W. Walker, and E. W. Dean, "The S.I.L. Viscosimeter," *Ind. and Eng. Chemistry*, Vol. 13, p. 346 (1941).

E. H. Zeitfuchs, "An Accurate Viscosimeter for Refinery Control Laboratories," *Proceedings* Am. Petroleum Inst., Vol. 20 (III), (1939).

Section 3.—Change to read as follows:

3. (*a*) Two procedures for determining kinematic viscosity by means of apparatus conforming to the requirements given in Section 2 appear in the Appendix. Any other procedure may be used provided it conforms to the same requirements.

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[3] These methods were accepted as tentative by the Society and appear in the 1946 Book of A.S.T.M. Standards, Part III-A.

[4] 1944 Book of A.S.T.M. Standards, Part III.

(*b*) For tests at temperatures below the dew point of the surrounding atmosphere it is necessary to prevent moisture from entering the apparatus and affecting the results.

New Section.—Add a new Section 5 to read as follows:

5. *Precision.*—Results should not differ from the mean by more than the following:

	Repeatability, One Operator and Apparatus	**Reproducibility,** Different Operators and Apparatus
Test temperature below 32 F:		
All oils	0.5	1.0
Test temperature 32 F. or above:		
Transparent oils	0.1	0.2
Opaque oils	0.3	0.5

Section A1 (*b*): ***Thermometers.***—Number the present note as Note 1 and add the following as a new Note 2:

NOTE 2.—Broad specifications for thermometers for temperatures below 0 F. are under consideration at this time. In the meantime, it is recommended that a mercury-thallium alloy thermometer (43 F – 45 T) prescribed in A.S.T.M. Specifications E 1 – 46 T,[6] resistance thermometer, or a multiple-junction thermocouple be used.

Section A1 (*b*). —Include thermometers for test temperatures of 68, 70, 77, 122, 140, and 180 F.

Section A1 (*c*).—Change the fourth sentence to read as follows: "The bath shall be regulated so that the variation in the thermometer reading does not exceed 0.05 F. (0.03 C.) for test temperatures of 32 F. or above, and 0.10 F. (0.06 C.) for test temperatures below 32 F.

Sections A2 (*d*) *and A6* (*f*).—Make the following changes in these two sections:

Change the first sentence to read as follows: "The bath shall be maintained at the temperature of test within plus or minus 0.025 F. for test temperatures of 32 F. or above, and within plus or minus 0.05 F. for test temperatures below 32 F. (See Note, Section A1 (*b*))."

Change the last sentence to read as follows: "The minimum time required is 5 min. for tests at 50 to 100 F. (10 to 37.78 C.) and 10 min. for tests at temperatures outside this range."

Add the following paragraph and note:

If the temperature of test is below the dew point of the surrounding atmosphere, water may condense in the viscosimeter and give erratic results. To avoid this, attach small, loosely packed drying tubes (Note) to all openings of the viscosimeter. If the sample of oil is saturated with water at atmospheric temperatures some of this water may separate in the form of a haze when the sample is cooled. Accurate viscosity determinations cannot be made on a hazy sample, consequently it is necessary to remove sufficient water so that the sample will be clear at the temperature of test. Excess moisture can be removed by warming sample of oil in a beaker to 150 to 180 F. (65.56 to 82.22 C.) while stirring with a glass rod for 5 or 10 min. and then fanning the top of the beaker to remove condensed moisture.

NOTE.—At very low temperatures considerable moisture may be absorbed by the drying tube. This may increase the resistance to flow of air through the drying tube thereby affecting the rate of flow of oil through the capillary of the viscosimeter. This may be avoided by connecting a large-bore stopcock from the end of viscosimeter tube 1 to the end of viscosimeter tube 2 between the viscosimeter and the drying tubes in the form of an H, so that air movement during the test will be through the open stopcock and not through the drying tubes.

Footnote 7.—Change the following items to read as follows:

Vol._2 = the total volume of liquid in the viscosimeter in cubic centimeters at T_2.
Vol._1 = the total volume of liquid in the viscosimeter in cubic centimeters at T_1.
H = the fluid head in centimeters; for viscosimeters shown in Fig. 2, this is approximately 9.0 cm., and

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[6] 1946 Book of A.S.T.M. Standards, Part III-A.

d = the inside diameter of the lower reservoir in centimeters; for viscosimeters shown in Fig. 2, this is approximately 3.0 cm.

Tentative Method of Test for Sediment in Fuel Oil by Extraction (**D 473 - 38 T**).[4] —Subcommittee XIV on Water and Sediment (C. F. Ramey, chairman) recommends the following revisions:

Section 2 (a).—Add the following phrase at the end of this section: "... or a wide-mouth Erlenmeyer flask of approximately 500-ml. capacity."

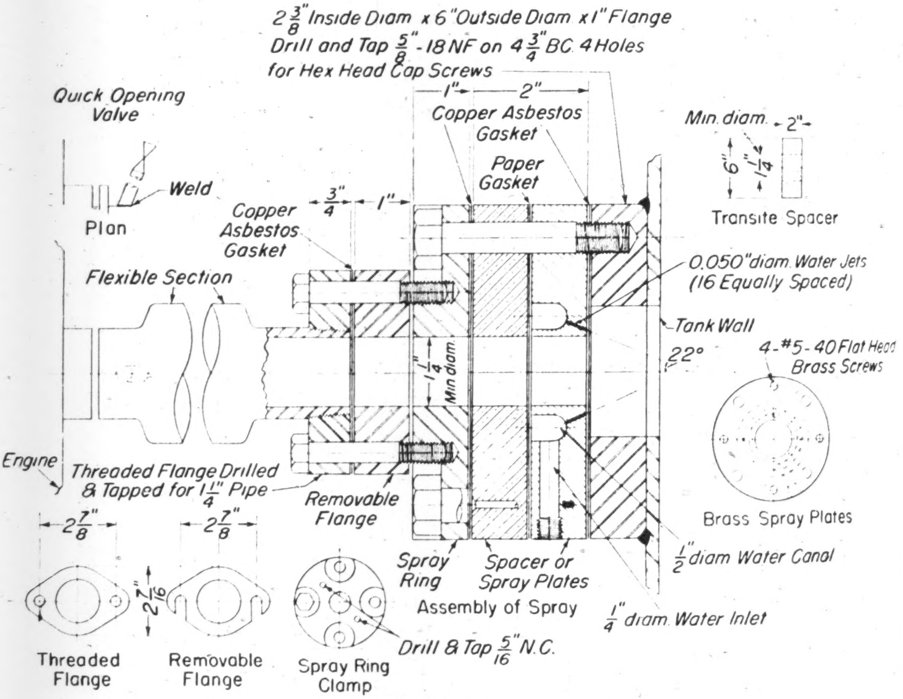

2 3/8" Inside Diam x 6" Outside Diam x 1" Flange
Drill and Tap 5/8"-18 NF on 4 3/4" BC 4 Holes
for Hex Head Cap Screws
Quick Opening
Valve
Weld
Plan
1"
2"
Copper Asbestos
Gasket
Paper
Gasket
Min. diam. 2"
6"
1/4"
Transite Spacer
3/4"
1"
Copper
Asbestos
Gasket
Flexible Section
0.050" diam. Water Jets
(16 Equally Spaced)
Tank Wall
22°
1 1/4" Min diam.
4-#5-40 Flat Head
Brass Screws
Engine
Threaded Flange Drilled
& Tapped for 1 1/4" Pipe
Removable
Flange
Brass Spray Plates
2 7/8"
2 7/8"
2 7/16"
Spray
Ring
Spacer or
Spray Plates
1/2" diam Water Canal
Assembly of Spray
1/4" diam. Water Inlet
Drill & Tap 5/16" N.C.
Threaded
Flange
Removable
Flange
Spray Ring
Clamp

FIG. 3.—Flexible Pipe and Spacer.

Section 2 (b).—Delete the present statement and Fig. 1, and replace with the following: "(*b*) *Condenser*.—Underwriters' form, comprising a block-tin coil approximately 1 in. in diameter and 2 in. in length, attached to and with the ends projecting through a lid of sufficient diameter to cover the neck of the flask."

Section 3.—To provide a more definite specification, change "90 per cent benzol" in the 17th line, to read "Industrial 90 Benzene (A.S.T.M. Specifications D 837).[4]"

Tentative Method of Test for Aniline Point and Mixed Aniline Point of Petroleum Products (**D 611 - 44 T**).[4]—Subcommittee XVIII on Aniline Point (Eugene Ayres, chairman) recommends the addition of the following note to Section 1 (*b*):

NOTE.—Mixed aniline points determined with *n*-heptane diluent will differ from those determined on the same product using various 60 C. aniline-point naphtha diluents. In general, the values obtained with *n*-heptane as diluent have been observed to be several degrees higher than those obtained with 60 C. aniline-point naphtha diluents. The difference depends both upon the nature of the product being tested and the nature of the particular 60 C. aniline-point naphtha employed as diluent.

Tentative Method of Test for Knock Characteristics of Aviation Fuels (**D 614 - 44 T**).[4]—Technical Committee A on

Gasoline (C. B. Veal, chairman) recommends a revision of this method as published in the 1945 Report of Technical Committee A,[6] which covers changes in the exhaust system specifications. It is also recommended that Figures 2 and 3 of the revision as published last year be replaced by the accompanying Figs. 3 and 4.

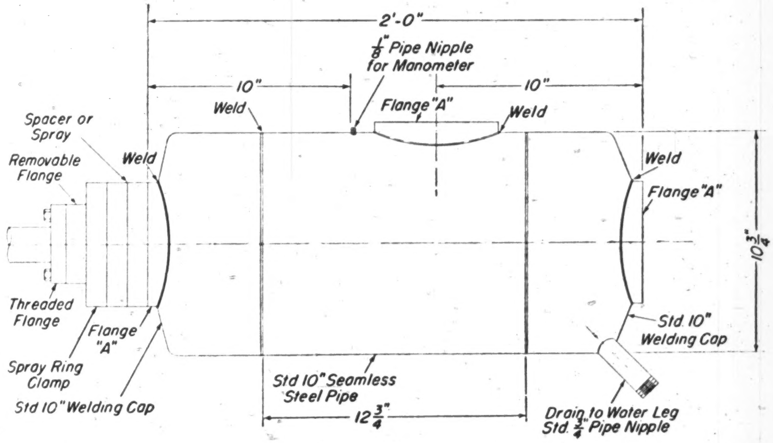
Tentative Method of Test for Acid and Base Numbers of Petroleum Products by Color-Indicator Titration (**D 663 – 44 T**).[4]—Subcommittee XIII on Neutralization Number and Saponification (H. P. Ferguson, chairman) recommends a revision of this method as appended hereto,[7] the changes being essentially editorial.

Tentative Method of Test for Acid and Base Numbers of Petroleum Products by Electrometric Titration (**D 664 – 44 T**).[4]—Subcommittee XIII also recommends a revision of this method as appended hereto,[7] the changes being essentially editorial.

Tentative Method of Test for Rust-Preventing Characteristics of Steam-Turbine Oil in the Presence of Water (**D 665 – 44 T**).[4]—Technical Committee C on Turbine Oils (F. C. Linn, chairman) recommends the following revisions:

Section 3 (*a*).—Add the following sentence to the end of this section: "The bath shall have a cover with holes to accommodate the test beakers."

Section 5 (*a*).—After the fourth sentence, add the following sentences: "The beaker shall be inserted into a hole of the bath cover and suspended in the hole with the beaker rim resting on the bath cover. The oil level in the bath shall not be below the oil level in the test beaker."


2'-0"
1/8" Pipe Nipple for Manometer
10"
10"
Weld
Flange "A"
Weld
Spacer or Spray
Removable Flange
Weld
Weld
Flange "A"
10 3/4"
Threaded Flange
Flange "A"
Std. 10" Welding Cap
Spray Ring Clamp
Std 10" Welding Cap
Std 10" Seamless Steel Pipe
12 3/4"
Drain to Water Leg Std. 3/4" Pipe Nipple

NOTE.—Tank to be lagged if water injection not used. Flange "A" 2 3/8 in. in inside diameter by 6 in. in outside diameter by 5/8 in. flange. Drill and tap 4 holes on 4 3/4 in. bolt circle for 5/8 in. hexagonal-head cap screws.

FIG. 4.—Exhaust System Surge Tank.

Tentative Method of Analysis of Petroleum Sulfonates (**D 855 – 45 T**).[8]—Subcommittee XXIV on Petroleum Sulfonates (V. F. Beyer, chairman) recommends this method be revised as appended hereto.[7]

III. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions in eight standard methods, as set forth below, and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these modifications may be referred to letter ballot of the Society.

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[6] See special compilation "A.S.T.M. Standards on Petroleum Products and Lubricants," September, 1945, p. 6; also *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 233 (1945).

[7] These revised methods were accepted by the Society and appear in the 1946 Book of A.S.T.M. Standards, Part III-A.

[8] 1945 Supplement to Book of A.S.T.M. Standards, Part III.

Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (**D 86 - 45**).[8] —The following addition is recommended by Subcommittee VIII on Distillation (E. W. Dean, chairman):

Section 2 (i).—As recommended by Technical Committee XII on Laboratory Apparatus, of Committee E-1 on Methods of Testing, add the following note:

NOTE.—Cylinders lacking the bottom 1-ml. line will not be rejected.

Standard Method of Test for Flash and Fire Points by Means of Open Cup (**D 92 - 45**).[8]—The following addition is recommended by Subcommittee XII on Flash Point (C. F. Ramey, chairman):

Section 7.—After "Reproducibility . . . 10 F." insert the following:

Results shall not differ from each other by more than the following on oils having a flash or fire point above 550 F.:

Repeatability (one operator and apparatus)	10 F.
Reproducibility (different operators and apparatus)	15 F.

Standard Method of Test for Flash Point by Means of the Pensky-Martens Closed Tester (**D 93 - 42**).[4]—The following revision is also recommended by Subcommittee XII:

Section 6.—Delete this present section on reproducibility of results, and substitute the following:

6. ***Precision.*** Results shall not differ from each other by more than the following on oils having a flash point up to and including 200 F.:

Repeatability (one operator and apparatus)	2 F.
Reproducibility (different operators and apparatus)	4 F.

Results shall not differ from each other by more than the following on oils having a flash point above 220 F.:

Repeatability (one operator and apparatus)	5 F.
Reproducibility (different operators and apparatus)	10 F.

Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (**D 95 - 40**).[4]—The following revisions are recommended by Subcommittee XIV on Water and Sediment (C. F. Ramey, chairman):

Section 2 (c).—Change "having an approximate capacity of 500 ml." to read "having a capacity of at least 500 ml."

Fig. 1.—Remove the flask measurement shown in Fig. 1 (*b*).

Section 2 (f).—Change to read as follows: (*f*) *Trap.*—"The trap shall be made of well-annealed glass constructed in accordance with Fig. 1 (*c*) and shall be graduated in 0.1-ml. divisions from 0 to 2 ml., and 0.2-ml. divisions from 2 to 10 ml. The error of the indicated capacity from 0 to 2 ml. shall not be greater than plus or minus 0.05 ml. and from 2 to 10 ml. it shall not exceed plus or minus 0.10 ml."

Section 5 (b).—In line 3 change "tight-fitting corks" to read "tight-fitting stoppers or ground-glass joints."

Standard Method of Test for Water and Sediment in Petroleum Products by Means of Centrifuge (**D 96 - 40**).[4]—The following revision is also recommended by Subcommittee XIV:

Section 4 (a).—Change "90 per cent benzol" in the first sentence to read "Industrial 90 Benzene (A.S.T.M. Specifications D 837).[6]"

Standard Method of Test for Carbon Residue of Petroleum Products (Conradson Carbon Residue) (**D 189 - 41**).[4]—The following addition is recommended by Subcommittee XXIII on Carbon Residue (L. C. Beard, Jr., chairman):

Section 1.—Add the following note:

NOTE.—The committee responsible for the development of this method urges that its use be discontinued and that instead the more expedient Ramsbottom Carbon Residue Test (A.S.T.M. Method D 524) be followed.

Standard Method of Test for Knock Characteristics of Motor Fuels (**D 357 - 45**).[8]—On the recommendation of Technical Committee A, it is proposed to revise these methods to include the changes in the mixture heater assembly and in the exhaust system specifications as published in the 1945 Report of Technical Committee A.[6] It is also recommended that Figures 2 and 3 of the revision as published last year be replaced by the accompanying Figs. 3 and 4. In addition, the following changes are also recommended:

Section 9.—Change the first paragraph to read as follows:

> 9. Inspection shall be made to see that the contact points are smooth, that all connections are tight, and that the diaphragm is gas-tight (a test under 200-psi. gage pressure is sufficient). After adjustment of the clearance between the bouncing pin and the diaphragm according to the manufacturer's instructions the final adjustment of spring tensions and contact point gap shall conform to both of the following requirements:

Section 10 (a).—Change the last sentence to read as follows:

> This is purely an engine check, and the bouncing-pin spring tensions and contact-point gap setting found to give the above match need not necessarily be used in subsequent tests. The clearance between the bouncing pin and the diaphragm, however, shall not be altered between the rating of standardization and test fuels.

Section A9.—Change the first five sentences and footnote 17 to read as follows:

> Knock intensity is measured by means of a bouncing pin, in conjunction with a knockmeter. The adjustable-leaf shoulder-suspended bouncing pin is the preferred type.[a] It consists of a cylindrical steel pin 0.218 ± 0.001 in. in diameter, to the upper end of which is fitted a fiber tip, the combined length of pin and tip being 7.000 ± 0.015 in. The pin is maintained in a vertical position on the engine by two bushings in a hollow barrel which is closed at the bottom by a spring-steel diaphragm 0.543 ± 0.003 in. in diameter, and 0.015 ± 0.0005 in. in thickness, locked in with a hollow nut. A shoulder bushing is threaded into the lower crossbar, and has attached to it a dial graduated in 25 equal divisions. Rotation of the dial and bushing through one full division results in its vertical displacement by 0.001 in. A shoulder on the fiber tip of the bouncing pin rests on the upper bushing dial, thus suspending the pin and providing an adjustable clearance between the lower end of the pin and the spring steel diaphragm in the bottom of the barrel.

> [a] The use of the bouncing pin described in the Tentative Method of Test for Knock Characteristics of Motor Fuels (A.S.T.M Designation: D 357 – 33 T), 1933 Book of A.S.T.M Tentative Standards, p. 540, or Tentative Method of Test for Knock Characteristics of Motor Fuels (A.S.T.M. Designation: D 357 – 39 T), 1939 Book of A.S.T.M. Tentative Standards, p. 170, is permissible.

Standard Method of Test for Gum Content of Gasoline (**D 381 – 44**).[4]—Technical Committee A also recommends that this method be revised as appended hereto.[7]

IV. TENTATIVE REVISION OF STANDARD

Standard Method of Test for Burning Quality of Kerosine Oils (**D 187 – 39**).[4]—Subcommittee XIX on Illuminating Oils (E. W. Dean, chairman) recommends that the Emergency Alternate Provisions EA – D 187[4] be published as a tentative revision of this standard.

V. ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following two tentatives be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Method of Test for Ash Content of Petroleum Oils (**D 482 – 43 T**),[4] on recommendation of Subcommittee XIV.

Tentative Method of Test for Oxidation Stability of Gasoline (**D 525 – 42 T**),[4] revised as appended hereto,[7] on recommendation of Technical Committee A.

VI. EDITORIAL CHANGES IN STANDARDS[9]

Standard Method of Test for Sulfur in Petroleum Oils by Bomb Method (**D 129 – 44**).[4]—Subcommittee VII on Sulfur Determination (H. M. Hancock, chairman) has recommended as an editorial change the addition of the following note to Section 1 on Scope:

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[9] **These recommendations were added to the report by action of Committee D-2 at its June meeting.**

NOTE.—For the determination of sulfur in lubricating oils containing additives and in additive concentrates, use A.S.T.M. Method D 894.[5]

Standard Method of Test for Acid Heat of Gasoline (**D 481 – 39**).[4]—Section B of Subcommittee XXV has recommended as an editorial change the addition of the following note to Section I on Scope:

NOTE.—A chemical method such as bromine titration or other suitable method is preferred to the acid heat method for determining reactive unsaturation. It is, therefore, recommended that specifications be written in terms of olefin content rather than acid heat. It is anticipated that the acid heat method will eventually be discontinued when it is no longer widely used in specifications.

VII. EMERGENCY STANDARDS

The committee recommends that the Emergency Method of Test for Color of U.S. Army Motor Fuel (All-Purpose) by Means of an A.S.T.M. Color Standard (**ES – 32**),[4] be continued, on the recommendation of Subcommittee VI, with the addition of the following note:

NOTE.—The A.S.T.M. postwar policy is to discontinue Emergency Standards. At the specific request of the Office of the Chief of Ordnance, U. S. War Department, this Emergency Method is being retained temporarily.

The committee recommends that the following five Emergency Standards be discontinued:

Emergency Methods:

Test for Isopentane and Benzene Insolubles in Used Lubricating Oils (ES – 42),

Test for Sulfated Residue from New Lubricating Oils (ES – 43a). This method in revised form is being submitted for publication as Tentative.

Test for Olefins, Aromatics, Paraffins, and Naphthenes in Aviation Gasoline (Without Distillation into Fractions) (ES – 45a). This

TABLE II.—ANALYSIS OF LETTER BALLOT VOTE

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE METHODS			
Test for Sulfated Residue from New Lubricating Oils	50	0	12
Olefins and Aromatics in Petroleum Distillates	51	0	11
Oxidation Stability of Aviation Gasoline (Potential Gum Method)	47	2	13
II. REVISION OF TENTATIVE METHODS			
Sulfur in Petroleum Oils by Lamp Method (D 90 – 41 T)	51	0	11
Kinematic Viscosity (D 445 – 42 T)	57	0	5
Sediment in Fuel Oil by Extraction (D 473 – 38 T)	47	2	13
Aniline Point and Mixed Aniline Point of Petroleum Products (D 611 – 44 T)	51	0	11
Knock Characteristics of Aviation Fuels (D 614 – 44 T)	43	0	19
Acid and Base Numbers of Petroleum Products by Color-Indicator Titration (D 663 – 44 T)	54	2	7
Acid and Base Numbers of Petroleum Products by Electrometric Titration (D 664 – 44 T)	53	0	10
Rust-Preventing Characteristics of Steam-Turbine Oil in the Presence of Water (D 665 – 44 T)	51	0	12
Analysis of Petroleum Sulfonates (D 855 – 45 T)	42	0	19
III. REVISIONS OF STANDARDS, IMMEDIATE ADOPTION			
Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (D 86 – 45)	55	0	7
Flash and Fire Points by Means of Open Cup (D 92 – 45)	58	0	5
Flash Point by Means of the Pensky-Martens Closed Tester (D 93 – 42)	56	0	6
Water in Petroleum Products and Other Bituminous Materials (D 95 – 40)	55	0	8
Water and Sediment in Petroleum Products by Means of Centrifuge (D 96 – 40)	54	1	8
Carbon Residue of Petroleum Products (Conradson Carbon Residue) (D 189 – 41)	50	1	13
Knock Characteristics of Motor Fuels (D 357 – 45)	43	0	19
Gum Content of Gasoline (D 381 – 44)	49	0	13
IV. TENTATIVE REVISION OF STANDARD			
Test for Burning Quality of Kerosine Oils (D 187 – 39)	44	0	17
V. ADOPTION OF TENTATIVES AS STANDARD			
Ash Content of Petroleum Oils (D 482 – 43 T)	51	0	9
Oxidation Stability of Gasoline (D 525 – 42 T)	50	1	12

method in revised form is being submitted for publication as tentative.

Test for Benzene, Toluene, and Higher-Boiling Aromatics in Aviation Gasoline (ES – 46), and

Test for Determination of Xylidine (ES – 47).

The committee recommends that the Emergency Alternate Provisions in Standard Method of Test for Sulfur in Petroleum Oils by Bomb Method (EA – D 129) be withdrawn. The Emergency Alternate Provisions EA – D 187 in Standard Method of Test for Burning Quality of Kerosine Oils (D 187 – 39) is to be withdrawn from this status and published as a tentative revision of Standard D 187, as mentioned earlier in this report.

VIII. TENTATIVES CONTINUED AS TENTATIVE

The committee recommended that the following specifications and methods, which have been published as tentative for two years or longer without revision, be continued as tentative because revisions are under consideration or in prospect:

Tentative Specifications for:

Fuel Oils (D 396 – 39 T),

Gasoline (D 439 – 40 T),

Tentative Methods of Test for:

Consistency of Lubricating Greases and Petrolatum (D 217 – 44 T),

Ignition Quality of Diesel Fuels (D 613 – 43 T),

Chlorine in Lubricating Oils by Bomb Method (D 808 – 44 T),

Chemical Analysis for Phosphorus in Lubricating Oils (D 809 – 44 T),

Chemical Analysis for Lead, Copper, and Iron in Lubricating Oils (D 810 – 44 T), and

Chemical Analysis for Metals in Lubricating Oils (D 811 – 44 T).

IX. REAFFIRMATION OF STANDARDS

The following standards have been published for six years or longer without revision, but are considered by the committee to be in accord with present practice and to require no revision:

Test for Flash Point by Means of the Tag Closed Tester (D 56 – 36),

Test for Precipitation Number of Lubricating Oils (D 91 – 40),

Test for Cloud and Pour Points (D 97 – 39),

Test for Melting Point of Petrolatum (D 127 – 30),

Methods of Analysis of Grease (D 128 – 40),

Test for Detection of Free Sulfur and Corrosive Sulfur Compounds in Gasoline (D 130 – 30),

Test for Color of Refined Petroleum Oil by Means of Saybolt Chromometer (D 156 – 38),

Test for Steam Emulsion of Lubricating Oils (D 157 – 36),

Volume Correction Table for Petroleum Oils (D 206 – 36),

Test for Distillation of Natural Gasoline (D 216 – 40),

Test for Burning Quality of Long-Time Burning Oil for Railway Use (D 219 – 36),

Test for Burning Quality of Mineral Seal Oil (D 239 – 30),

Test for Thermal Value of Fuel Oil (D 240 – 39),

Methods of Sampling Petroleum and Petroleum Products (D 270 – 33),

Test for Autogenous Ignition Temperatures of Petroleum Products (D 286 – 30),

Test for Gravity of Petroleum and Petroleum Products by Means of the Hydrometer (D 287 – 39),

Definitions of Terms Relating to Petroleum (D 288 – 39),

Test for Dilution of Crankcase Oils (D 322 – 35),

Conversion of Kinematic Viscosity to Saybolt Universal Viscosity (D 446 – 39),

Test for Unsulfonated Residue of Plant Spray Oils (D 483 – 40), and

Specifications for Stoddard Solvent (D 484 – 40)

X. CONTINUATION OF STANDARD

The committee recommends that the Standard Method of Test for Acid Heat of Gasoline (D 481 – 39), which also has been published for over six years without revision, be continued but that no note of reaffirmation be printed thereon.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 80 voting members, with the results shown in Table II.

The election of officers for the ensuing term of two years resulted in the election of the following:

Chairman, C. Dantsizen.
Vice-Chairman, T. G. Delbridge.
Secretary, W. T. Gunn.

This report has been submitted to letter ballot of the committee, which consists of 80 voting members; 63 members returned their ballots, of whom 58 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

T. A. BOYD,
Chairman.

D. V. STROOP,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-2 presented to the Administrative Committee on Standards the following recommendations:

New Tentative Methods for:

Test for Normal Pentane and Benzene Insolubles in Used Lubricating Oils (D 893 – 46 T), and

Test for Sulfur in Lubricating Oils Containing Additives and in Additive Concentrates by Bomb Method (D 894 – 46 T).

These recommendations were accepted by the Standards Committee on November 6, 1946, and the new tentatives appear in the 1946 Book of A.S.T.M. Standards, Part III-A.

On December 5, 1946, the Standards Committee approved the new Tentative Method of Test for Foaming Characteristics of Viscous Petroleum Oils (D 892 – 46 T) which also appears in the 1946 Book of A.S.T.M. Standards, Part III-A.

The emergency provisions listed in the report were discontinued by action of the Standards Committee on September 9, 1946.

APPENDIX I

PROPOSED METHOD OF TEST FOR SULFATED RESIDUE OF LUBRICATING OILS[1]

INDIRECT ACID-OXIDATION METHOD

This is a proposed method and is published as information only.[2] Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method is intended for the determination of the quantity of sulfated residue obtainable from new and used lubricating oils and from lubricating oil additive concentrates. It may be used to indicate the concentration of known metal-containing additives in new oils. Sulfur, bromine, and chlorine do not interfere; phosphorus, when present in concentrations of 0.1 per cent or more, tends to interfere by remaining partially in the residue.

NOTE 1.—A.S.T.M. Method D 482, Test for Ash Content of Petroleum Oils,[3] does not generally give comparable or related results when applied to additive-type oils and, in general, is not recommended for such oils.

Outline of Method

2. The sample is ignited and burned until only ash and carbon remain. After cooling, the charred residue is treated with sulfuric acid, the carbon completely removed by oxidation with nitric acid, and the excess acid removed by evaporation. The residue is then ignited at 775 C. and weighed.

Apparatus

3. (*a*) *Electric Muffle Furnace.*—The furnace shall be capable of operating over a variable temperature range of 200 to 800 C. and of maintaining a temperature of 775 ± 25 C.

(*b*) *Silica Beakers.*—Beakers of 100-ml. and 250-ml. capacities, made of fused silica.[4]

(*c*) *Porcelain Crucible*, 30-ml. capacity

Reagents

4. (*a*) *Sulfuric Acid, c.p. (sp. gr. 1.84).*

(*b*) *Nitric Acid, c.p. (sp. gr. 1.42).*

Sample

5. (*a*) Heat the oil sample to 60 ± 10 C. and agitate it in the original container until all sediment is homogeneously suspended in the oil. If the original container is of opaque material, or if it is more than three-fourths full, transfer the entire sample (including the sediment) to a clear glass bottle having a capacity at least one third greater than the volume of the sample. Transfer all traces of sediment from the original container to the bottle by violent agitation of portions of the sample in the original container. After complete suspension of all sediment, strain the sample or a convenient aliquot through a 100-mesh screen for the removal of large contaminating particles. Discard the residue remaining on the screen.

(*b*) Heat and thoroughly mix the strained oil or aliquot before taking *each* portion of the sample for test. Homogeneity will be indicated by the precision of the analyses.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

[2] Published as information, June, 1946

[3] 1946 Book of A.S.T.M. Standards, Part III-A.

[4] Vycor beakers are also satisfactory.

Procedure for Burning and Igniting in Beaker

6. (*a*) Ignite a 100-ml. silica beaker at 700 to 800 C. for 10 min. or more, cool to room temperature in a desiccator, and weigh to the nearest 0.1 mg. within 20 to 30 min. after cooling. Weigh into the beaker 10 ± 0.1 g. of well-mixed sample. Heat the beaker with a bunsen burner until the contents ignite and burn readily, then move the beaker and flaming contents to a hot plate. Maintain the hot plate at such a temperature that the sample continues to burn at a uniform and moderate rate, leaving only ash and carbon when the burning ceases.

(*b*) Cool the beaker, add 5 to 10 ml. of H_2SO_4 (sp. gr. 1.84), cover the beaker with a watch glass, and heat to white fumes. Add HNO_3 (sp. gr. 1.42), dropwise, down the sides of the beaker, until the solution is clear and the beaker is free from carbonaceous matter. Cool, thoroughly wash any residue from the watch glass into the beaker, and discard the watch glass. Cautiously evaporate the solution to dryness. If the residue is not white in color, moisten with H_2SO_4 (sp. gr. 1.84), heat to white fumes, add HNO_3 (sp. gr. 1.42) dropwise to complete the oxidation of the organic matter, and again evaporate to dryness. Cool the furnace to 300 C. or lower and place the beaker in the furnace. Gradually increase the temperature to 775 ± 25 C. and maintain at that temperature for 25 to 30 min. Cool the beaker to room temperature in a desiccator and weigh to the nearest 0.1 mg. Reignite the beaker at 775 C. for 10 to 20 min., cool, and reweigh. Repeat the heating and weighing until consecutive weighings differ by 1.0 mg. or less.

NOTE 2.—For greatest accuracy with samples containing less than 0.5 per cent residue, it is desirable to use a similar beaker as a counterpoise in weighing the beaker and to take care that the treatment of the beaker prior to weighing is always the same.

Procedure for Burning in Crucible and Igniting in Beaker

7. (*a*) Into a clean, dry, 30-ml. crucible, weigh 10 ± 0.1 g. of well-mixed sample. Heat the crucible with a bunsen burner until the contents ignite and burn readily, then move the crucible and flaming contents to a hot plate. Maintain the hot plate at such a temperature that the sample continues to burn at a uniform and moderate rate, leaving only ash and carbon when the burning ceases.

(*b*) Ignite a 250-ml. silica beaker at 700 to 800 C. for 10 min. or more, cool to room temperature in a desiccator, and weigh to the nearest 0.1 mg. within 20 to 30 min. after cooling.

(*c*) Place the cool crucible and carbonaceous residue (Paragraph (*a*)) into the weighed 250-ml. silica beaker. Add 5 to 10 ml. of H_2SO_4 (sp. gr. 1.84), cover the beaker with a watch glass, and heat to white fumes. Add HNO_3 (sp. gr. 1.42) dropwise, to the fuming contents of the beaker until the solution and crucible are free from carbonaceous matter. Cool, thoroughly wash any residue from the crucible and watch glass into the beaker, and discard the watch glass and crucible. Cautiously evaporate the solution to dryness. If the residue is not white in color, moisten with H_2SO_4 (sp. gr. 1.84), heat to white fumes, add HNO_3 (sp. gr. 1.42) dropwise, to complete the oxidation of the organic matter, and again evaporate to dryness. Cool the furnace to 300 C. or lower and place the beaker in the furnace. Gradually increase the temperature to 775 ± 25 C. and maintain at that temperature for 25 to 30 min. Cool the beaker to room temperature in a desiccator and weigh to the nearest 0.1 mg. Reignite the beaker at 775 C. for 10 to 20 min., cool, and reweigh. Repeat the heating and weighing until consecutive weighings differ by 1.0 mg. or less.

Calculation

8. Calculate the sulfated residue of the sample as follows:

$$\text{Sulfated Residue, per cent} = \frac{R \times 100}{W}$$

where:

R = weight of residue in grams, and

W = weight of sample in grams.

Precision

9. Results should not differ from the mean by more than the following amounts:

Sulfated Residue, per cent	**Repeatability,** One Operator and Apparatus	**Reproducibility,** Different Operators and Apparatus
Below 1.0.......	0.03	0.05
1.0 to 8.0.......	0.05	0.08

APPENDIX II

PROPOSED METHOD OF TEST FOR PHOSPHORUS IN ORGANIC MATERIALS[1]

ACID-OXIDATION METHOD

This is a proposed method and is published as information only.[2] Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method is intended for the determination of total phosphorus in concentrations greater than 0.5 per cent in all types of organic compounds including lubricating oils, lubricating oil additives, and their concentrates. Common metals, sulfur, nitrogen, and chlorine do not interfere.

Outline of Method

2. Organic material in the sample is removed and the phosphorus is converted to phosphate ion by oxidation with sulfuric acid, nitric acid, and hydrogen peroxide. If necessary, the phosphate ion is separated from interfering metals by precipitating as ammonium molybdiphosphate in nitric acid solution. After an ammoniacal solution of the phosphate ion is obtained, the phosphorus is precipitated as magnesium ammonium phosphate, ignited, and weighed as magnesium pyrophosphate.

Apparatus

3. (*a*) *Digestion Rack.*—A digestion rack constructed to hold one or more 300-ml. Kjeldahl flasks at an angle of approximately 45 deg. in such a fashion that direct heat is applied only to the bottom of the flask over an area less than 2 in. in diameter, and such that the body and neck of the flask are insulated from the source of heat. Approximately three fourths of the neck of the flask should be cooled by air at atmospheric temperature, preferably by directing an air stream against the neck of the flask. A bunsen flame or high-capacity electric heater are suitable heat sources.

(*b*) *Electric Muffle Furnace.*—The furnace shall be capable of operating over a variable temperature range of 200 to 1100 C. and of maintaining a temperature of 1050 ± 50 C.

Reagents

4. (*a*) *Ammonium Hydroxide, c.p., (sp. gr. 0.90).*

(*b*) *Ammonium Hydroxide (10 per cent).*—To prepare 1 liter of solution, mix 380 ml. of c.p. NH_4OH (sp. gr. 0.90) with 620 ml. of distilled water.

(*c*) *Ammonium Hydroxide (1 per cent).*—To prepare 1 liter of solution, mix 40 ml. of c.p. NH_4OH (sp. gr. 0.90) with 960 ml. of distilled water.

(*d*) *Ammonium Nitrate, c.p.*—NH_4NO_3 crystals.

(*e*) *Ammonium Nitrate Solution (5 per cent).*—To prepare 1 liter, dissolve 50 g. of NH_4NO_3 in distilled water and dilute to 1 liter.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

[2] Published as information, June, 1946.

(*f*) *Hydrochloric Acid, c.p.* (*sp. gr. 1.18*).

(*g*) *Hydrogen Peroxide, c.p.* (*30 per cent*)

(*h*) *Magnesia Mixture.*—Dissolve 50 g. of $MgCl_2 \cdot 6H_2O$ and 100 g. of NH_4Cl in 500 ml. of distilled water, add a slight excess of NH_4OH, and allow to stand overnight. Filter, make the solution just acid with HCl, and dilute to 1 liter.

(*i*) *Molybdate Reagent.*—Dissolve 100 g. of $(NH_4)_2MoO_4$ in 400 ml. of water. Add 80 ml. of NH_4OH (sp. gr. 0.90) and filter if a precipitate appears. Mix 400 ml. of HNO_3 (sp. gr. 1.42) with 600 ml. of water. Prepare the ammonium molybdate reagent from these solutions immediately before use by slowly mixing one volume of the ammonium molybdate solution with two volumes of the diluted HNO_3, while stirring rapidly.

(*j*) *Nitric Acid, c.p.,* (*fuming, sp. gr. 1.5*).

(*k*) *Nitric Acid, c.p.,* (*sp. gr. 1.42*).

(*l*) *Nitric Acid* (*40 per cent*).—Prepare by mixing approximately equal volumes of c.p. HNO_3 (sp. gr. 1.42) and water.

(*m*) *Sulfuric Acid, c.p.* (*sp. gr. 1.84*).

Procedure for Samples Containing no Metals Other Than Alkali Metals

5. (*a*) Into a 300-ml. Kjeldahl flask, place 10 ml. of H_2SO_4 (sp. gr. 1.84) and 2 ml. of fuming HNO_3. Cool the flask and contents to ice temperature, and add to the flask a portion of sample of the following size:

Phosphorus Content, per cent	Approximate Weight of Sample, g.	Precision of Weighing, g.
Over 0.5 to 2	4	±0.01
Over 2 to 5	2	±0.005
Over 5 to 10	1	±0.003
Over 10 to 15	0.7	±0.002
Over 15 to 25	0.4	±0.001

Allow the sample to fall directly into the acid mixture (Note 1), and avoid getting any sample on the neck of the flask. Thoroughly mix the sample and acids by swirling.

NOTE 1.—In the case of the samples that react vigorously with HNO_3, it is desirable to add the sample to the flask containing only the H_2SO_4 and to add the fuming HNO_3 dropwise while mixing. However, it is necessary to add the 2 ml. of HNO_3 prior to heating the mixture for digestion.

(*b*) Place the flask on the digestion rack, insert a small funnel into the neck of the flask, and cool the neck of the flask (preferably by use of an air stream; see Note 2). Apply heat gradually to the bottom of the flask until fumes of H_2SO_4 are evolved strongly above the liquid in the flask. Cool the flask slightly, add 1 to 2 ml. of HNO_3 (sp. gr. 1.42) dropwise (Note 3) and again heat the flask to strong fumes of H_2SO_4. Repeat this oxidation with HNO_3 until nearly all of the organic material has been oxidized as evidenced by the light color of the fuming H_2SO_4 solution. Cool for several minutes, add dropwise 1 to 2 ml. of H_2O_2 (30 per cent), and again heat to fumes of H_2SO_4. Repeat until the fuming solution becomes pale yellow or colorless, indicating complete oxidation of the organic material.

NOTE 2.—The amount of air used to cool the neck of the flask will at times have to be reduced or even shut off to allow vapors and fumes to leave the flask and to allow the sample to come to fumes of H_2SO_4. However, this should not be done until the sample is in a well-decomposed state; the air stream should be turned on again each time before the addition of the HNO_3 or H_2O_2.

NOTE 3.—Unless the HNO_3 is added dropwise, it may force excessive amounts of vapor from the flask and lead to loss of phosphorus-containing fumes.

(*c*) Cool the flask, transfer the solution to a 400-ml. beaker, and wash the flask with small portions of distilled water until the volume of solution is approximately 100 ml. Boil the solution for 5 to 10 min., cool to near room temperature, and add NH_4OH (sp. gr. 0.90) until the solution is neutral to methyl red. Make the solution acid with HCl (sp. gr. 1.18) and add 1 ml. in excess.

(*d*) Add 20 ml. of magnesia mixture, slowly and while stirring, and cool the solution to below room temperature in an ice bath. Add NH_4OH (sp. gr. 0.90), slowly and while stirring constantly, until the solution is basic. Continue stirring until most of the precipitate has formed; then add 5 ml. of NH_4OH (sp. gr. 0.90) in excess. Allow the precipitate to stand overnight, filter through a weighed porcelain filter crucible of fine porosity, wash with NH_4OH (1 per cent), and dry in an oven. Place in a cool furnace, gradually raise the temperature to red heat, and ignite at 1050 ± 50 C. for 30 to 40 min. Repeat the ignition for similar periods until constant weight is reached.

NOTE 4.—For work of highest accuracy, it is generally necessary to test the precipitation technique on known inorganic samples. Reprecipitation sometimes aids in obtaining more accurate values.

Procedure for Samples Containing Metals Other Than Alkali Metals

6. (*a*) Decompose the sample as directed in Sections 5 (*a*) and (*b*).

(*b*) Cool, add 40 to 50 ml. of water, cool to room temperature, and filter the solution through a medium-texture, ashless paper. Collect the filtrate in a 500-ml., wide-mouth, glass-stoppered Erlenmeyer flask, and wash the Kjeldahl flask and filter paper thoroughly with water; discard the paper. Boil the solution for several minutes and cool to near room temperature. Add NH_4OH (sp. gr. 0.90) until the solution is neutral to methyl red, then add HNO_3 (40 per cent) until the color just changes to red. Concentrate or dilute the volume of the solution to approximately 150 ml.

(*c*) Add 15 g. of NH_4NO_3 crystals and swirl until dissolved. Adjust the temperature to 35 to 40 C. and add 120 ml. of freshly prepared molybdate reagent. Stopper the flask, shake vigorously for 4 to 6 min., and allow to stand for at least 2 hr. or preferably overnight. Filter the solution through a medium-texture, ashless paper. Wash the precipitate with NH_4NO_3 solution (5 per cent). Do not attempt to transfer all of the precipitate from the flask to the paper; however, reserve the flask for later treatment. Wash the precipitate several times with the wash solution but do not allow the stream of wash solution to strike the funnel above the edge of the paper as the precipitate has a tendency to creep.

(*d*) Place a clean 400-ml. beaker under the funnel and dissolve the precipitate through the paper into the beaker with NH_4OH (10 per cent). Use a little of the NH_4OH to dissolve any of the precipitate that remained in the flask set aside in Paragraph (*c*) and pour this solution through the paper. Wash the flask, funnel, and paper four times with hot water, once with NH_4OH (10 per cent), and once again with water. Discard any residue remaining on the paper. Evaporate the solution to a volume of 90 to 100 ml., make the solution acid with HCl (sp. gr. 1.18), and add 1 ml. in excess. Disregard any molybdiphosphate precipitate that may appear at this point.

(*e*) Add 10 ml. of magnesia mixture slowly, while stirring, and cool the solution to below room temperature in an ice bath. Add NH_4OH (sp. gr. 0.90) slowly, while stirring constantly, until the solution is basic. Continue stirring until most of the precipitate has formed, then add 5 ml. of NH_4OH (sp. gr. 0.90) in excess. Allow the precipitate to stand overnight, filter through a weighed porcelain filter crucible (fine porosity), wash with NH_4OH (1 per cent), and dry in an oven. Place in a cool furnace, gradually raise the temperature to red heat, and ignite at 1050 ± 50 C. for 30 to

40 min. Repeat the ignition for similar periods until constant weight is reached.

Calculation

7. Calculate the phosphorus content as follows:

$$\text{Phosphorus Content, per cent} = \frac{P \times 27.84}{W}$$

where:

P = weight of magnesium pyrophosphate in grams, and

W = weight of sample taken in grams.

Precision and Accuracy

8. (*a*) Results should not differ from the mean by more than the following amounts:

Phosphorus Content, per cent	**Repeatability,** One Operator and Apparatus	**Reproducibility,** Different Operators and Apparatus
0.5 to 5	0.02	0.04
Over 5 to 25	0.05	0.10

(*b*) The accuracy is of the same order of magnitude as the reproducibility.

APPENDIX III

PROPOSED METHOD OF TEST FOR AROMATIC HYDROCARBONS IN MIXTURES WITH NAPHTHENES AND PARAFFINS BY SILICA GEL ADSORPTION[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the Society, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method covers the test for the determination of aromatic hydrocarbons in mixtures with naphthenic and paraffinic hydrocarbons. The method is limited to materials boiling in the gasoline range containing not more than about 1 per cent of olefinic hydrocarbons.

Outline of Method[2]

2. The sample is entered onto the top of a column packed with powdered activated silica gel and, when all of the sample has been adsorbed on the column of gel, alcohol is added as an eluent. The alcohol causes desorption of the hydrocarbons, forcing them down the column. In this process of adsorption and desorption the hydrocarbons are separated according to their adsorbability so that the components issue from the bottom of the silica gel column in the following order: saturates, saturates plus aromatics, aromatics, aromatics plus alcohol, and alcohol. The percolate is collected in small fractions and the aromatic content of the sample is calculated from measured volumes and refractive indices of the fractions.

Apparatus

3. (*a*) *Adsorption Column*, as shown in Fig. 1. The column should be surrounded with a transparent protective shield when operating at pressures in excess of 10 psi.

(*b*) *Receivers* of 5- and 10-ml. capacities as shown in Fig. 2.

(*c*) *Sample Containers* of approximately 5-, 10-, and 25-ml. capacities. Numbered sample vials with molded plastic screw caps combined in sets, each set supported in a drilled wooden block, make a convenient arrangement.

(*d*) *Refractometer*, Abbe-type, capable of measuring refractive indices to plus or minus 0.0001 of a unit.

Materials

4. (*a*) *Silica Gel*, activated,[3] passing the sieve test shown in Table I. Test each lot of gel as follows: Percolate through the packed column (see Section 7) a mixture of 80 ml. of essentially pure 2,2,4-trimethylpentane and 20 ml. of essentially pure toluene and follow with alcohol as described in Section 8. Consider the lot as having satisfactory adsorptive properties if not more than 3 ml. of percolate of refractive index, n_D^{25}, between 1.3940 and 1.4890 is obtained.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1946.

[2] For a more complete discussion of this method, see B. J. Mair, "Separation and Determination of Aromatic and Monoolefin Hydrocarbons in Mixtures with Paraffins and Naphthenes by Adsorption," *Journal of Research*, Nat. Bureau Standards, Vol. 34, No. 5, 1945, p. 435.

[3] Silica gel, No. 22-08 from the Davison Chemical Corp., Baltimore, Md., is satisfactory.

(*b*) *Alcohol, Ethyl or Isopropyl,* c.p. absolute.

(*c*) *Toluene,* at least 99 per cent pure.

(*d*) *Isooctane,* 2,2,4-trimethylpentane at least 99 per cent pure.

(*e*) *Diluent,* an aromatic-free hydrocarbon or a mixture of hydrocarbons boiling in the gasoline range.

(*f*) *Gas,* compressed air, nitrogen, or carbon dioxide delivered to the top of the adsorption column at a regulated pressure of at least 20 psi. constant to plus or minus 1 psi.

TABLE I.—GEL SIEVE ACCEPTANCE TESTS.

Sieve Size[4]		Particles Passing Through Sieve, per cent
Microns	Designation[4]	
149	No. 100	95 min.
74	No. 200	80 min.
44	No. 325	25 max.

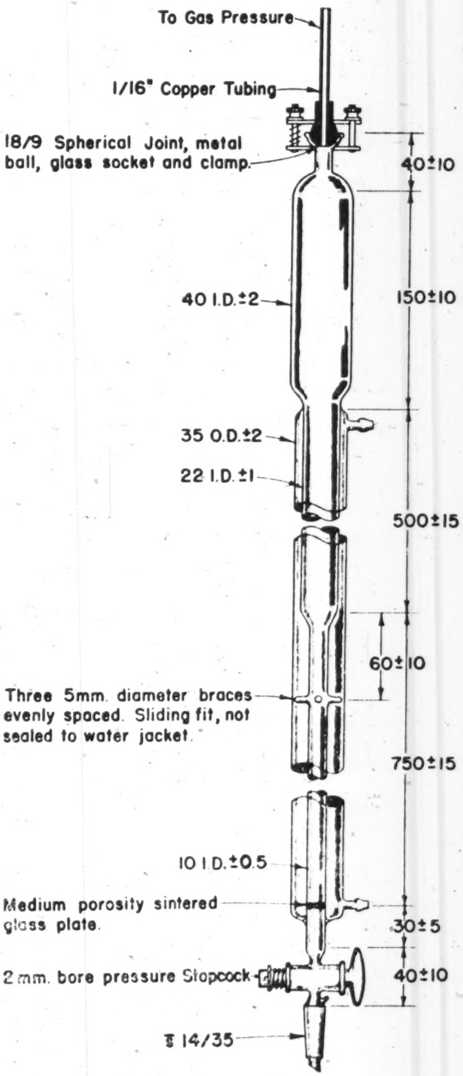
(*g*) *Stopcock Lubricant,* hydrocarbon insoluble.[5]

Standardization of Receivers

5. (*a*) By means of a 100-ml. pipette deliver 100 ml. of toluene (pipette need not be calibrated) to the dry, empty, adsorption column, permitting the toluene to drain down the sides of the column.

NOTE 1.—Lubricate only the stopcocks of the adsorption column and of the receiver with hydrocarbon insoluble lubricant.

(*b*) Admit toluene into the receiver until the liquid level reaches the 0.98-ml. mark. After a drainage period of 40 ±5 sec., estimate and record the volume to 0.002 ml. Open the receiver stopcock fully and permit the toluene to drain into a bottle for 40 ±5 sec. In this fashion fill the receiver with about one hundred 1-ml. portions (Note 2). It is essential that all the liquid be drained from the column and measured, but it is not essential that there be exactly 100 portions or that the last two or three portions be close to 1.00 ml.


To Gas Pressure
1/16" Copper Tubing
18/9 Spherical Joint, metal ball, glass socket and clamp.
40±10
150±10
40 I.D.±2
35 O.D.±2
22 I.D.±1
500±15
60±10
Three 5mm. diameter braces evenly spaced. Sliding fit, not sealed to water jacket.
750±15
10 I.D.±0.5
Medium porosity sintered glass plate.
30±5
2 mm. bore pressure Stopcock
40±10
$ 14/35

All dimensions in millimeters.

FIG. 1.—Adsorption Column.

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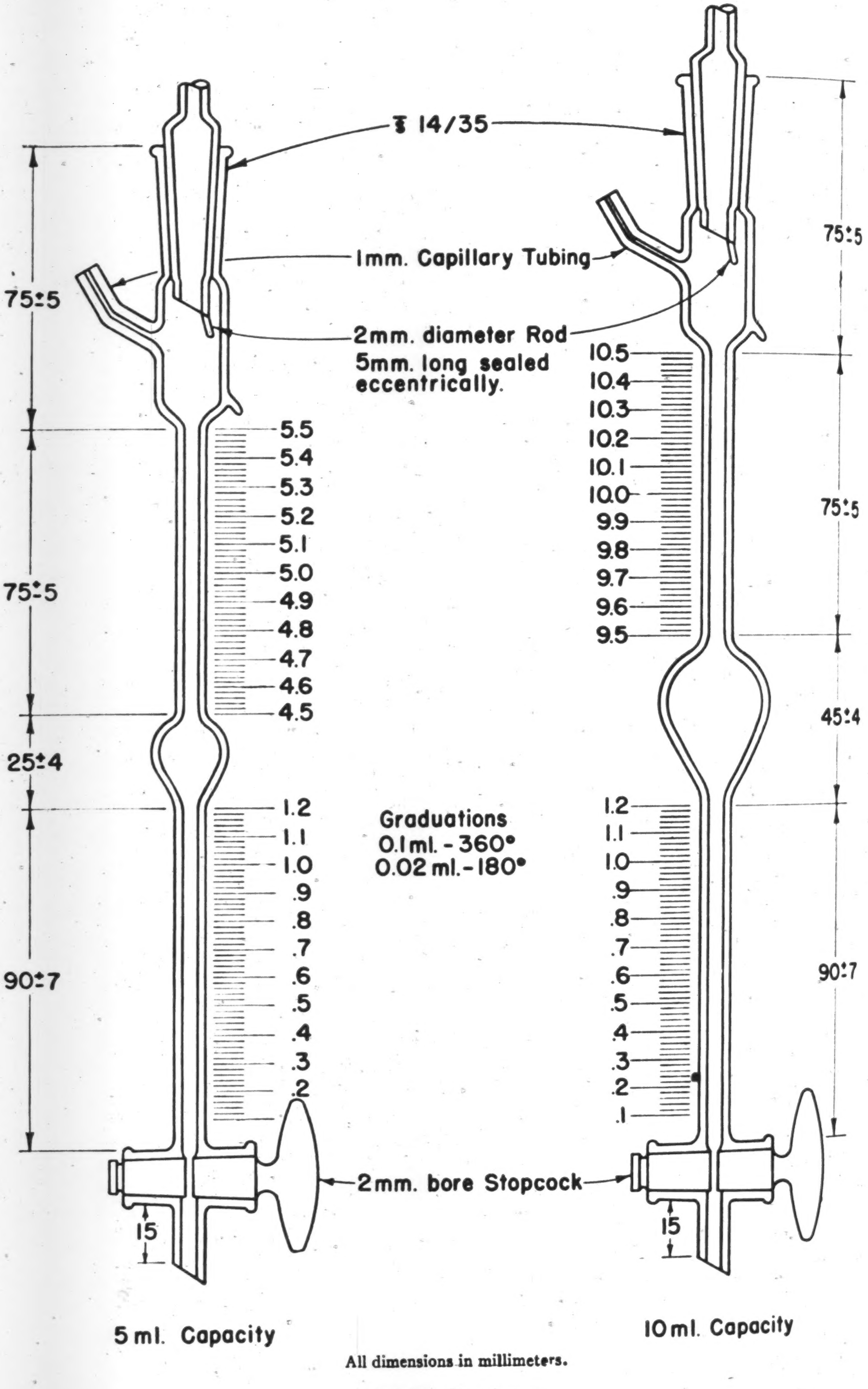
[4] Detailed requirements for these sieves are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), 1946 Book of A.S.T.M. Standards, Part III-A.

[5] For a suitable recipe, see T. P. Sager, "An Esterification Resin as a Ground Joint Lubricant," *Ind. and Engr. Chemistry,* Analytical Ed., Vol. 4, No. 4, 1932, p. 388.


Ŧ 14/35
1mm. Capillary Tubing
2mm. diameter Rod
5mm. long sealed
eccentrically.
75±5
75±5
25±4
90±7
75±5
75±5
45±4
90±7
5.5
5.4
5.3
5.2
5.1
5.0
4.9
4.8
4.7
4.6
4.5
1.2
1.1
1.0
.9
.8
.7
.6
.5
.4
.3
.2
10.5
10.4
10.3
10.2
10.1
10.0
9.9
9.8
9.7
9.6
9.5
1.2
1.1
1.0
.9
.8
.7
.6
.5
.4
.3
.2
.1
Graduations
0.1 ml. - 360°
0.02 ml. - 180°
2mm. bore Stopcock
15
15
5 ml. Capacity
10 ml. Capacity

All dimensions in millimeters.

FIG. 2.—Receivers.

(*c*) Dry the column and receiver, and add a second identical portion of toluene to the column as used in Paragraph (*a*), using the same pipette and identical filling technique. In the manner described in Paragraph (*b*), fill the receiver with ten 10-ml. portions (Note 2) or twenty 5-ml. portions, depending on the receiver used.

NOTE 2.—A temperature variation not greater than 3 C. can be tolerated during the standardization of any one mark without applying temperature corrections to the observed volumes.

(*d*) Calculate the volume correction factors (Note 3) to be used at about the 5- or 10-ml. mark from the following:

$$K_5 = \frac{V_5}{V_1}$$

$$K_{10} = \frac{V_{10}}{V_1}$$

where:

V_1, V_5, V_{10} = sum of volume readings at about the 1-, 5- or 10-ml. marks, respectively.

NOTE 3.—These are the factors by which the volumes delivered by the receivers at the 5- or 10-ml. marks must be multiplied to obtain the volume in units of the same size as the volume delivered by the corresponding receiver at the 1-ml. mark.

Test for Impurities in Silica Gel

6. (*a*) On each lot of silica gel, determine the gel-alcohol blank for isopropyl and ethyl alcohols by adding about 175 ml. of the alcohol to the column packed as described in Section 7. Force the alcohol through the adsorbent at a rate of descent of 7 to 13 mm. per min. (0.3 to 0.7 ml. per min.). Collect the first 5 ml. and measure its refractive index, n_D^{25}, to plus or minus 0.0001 of a unit.

NOTE 4.—Commercial silica gel contains a small amount of organic material desorbed by alcohol which appears in the fractions containing mixtures of aromatics and alcohol, thus giving an apparent aromatic content slightly greater than is actually present.

(*b*) The gel-alcohol blank, *C*, in milliliters, for ethyl alcohol is the difference between the measured refractive index of the first 5 ml. of alcohol collected and 1.3600, multiplied by 37. For isopropyl alcohol it is the difference between the refractive index of the 5 ml. collected and 1.3755, multiplied by 42.

NOTE 5.—The values 1.3600 and 1.3755 are those usually found for the sixth 1-ml. fraction of alcohol issuing from the column and are those used in the calculation (see Section 9(*c*) item (*4*)). Additional alcohol taken through the column would show a further decrease in refractive index approaching that of the pure alcohol asymptotically, at a value lower than the above by several units in the fourth decimal place.

Preparation of Apparatus

7. (*a*) Clean column and receiver with chromic-sulfuric acid, water, and acetone or alcohol, and dry them by evacuation (Note 6). Lightly lubricate the stopcocks (but not the spherical joint or the standard taper joint) with hydrocarbon-insoluble lubricant.

NOTE 6.—Clean the receivers with cleaning acid after each use and the column after about every third analysis.

(*b*) With the column clamped to its stand, open the stopcock at the bottom of the column and pour in from the top, through a funnel, silica gel to a point 20 to 30 mm. inside the reservoir. Immediately close the column stopcock and close the reservoir with a rubber stopper.

(*c*) Pack the silica gel by tapping throughout its entire length with a rubber-covered rod, or with an electric vibrator clamped to the middle portion of the water jacket, until the surface of the gel has dropped to a point 30 to 50 mm. below the reservoir and does not move more than 5 mm. during 5 min. of tapping or 0.5 min. of vibrating. Add more silica gel if the quantity proves insufficient. If not used immediately, leave the column closed to protect the gel from atmospheric moisture.

Procedure

8. (*a*) Pass tap water through the jacket; for samples containing appreciable quantities of material boiling below 40 C., circulate ice water. Attach a receiver of proper capacity (Table II) to the column and connect the vent of the receiver to an open drying tube to protect the gel which is very hygroscopic.

TABLE II.—SIZE OF SAMPLE FOR ANALYSIS.

Aromatics, per cent by volume	Sample, ml.	Diluent, ml.	Receiver[a] Capacity, ml.
0 to 15	125	0	10
15 to 20	100	0	10
20 to 30	70	0	10
30 to 40	50	0	5
40 to 50	40	0	5
50 to 80	30	0	5
80 to 100	25[b]	5[b]	5

[a] Do not change receivers during a test.

[b] To mix, transfer five 5-ml. portions of sample and one 5-ml. portion of diluent to a 50-ml. glass-stoppered flask or bottle and mix thoroughly by shaking. Use the same pipette for all measurements (calibration unnecessary). A convenient method of transfer is described in Section A2 (*c*) of the Appendix.

(*b*) Open the column stopcock. Pipette into the reservoir a quantity of sample dependent upon its aromatic content as indicated in Table II (Note 7), avoiding wetting of the sides of the reservoir. (No exact pipetting required.) Lubricate the spherical joint and fasten the screw clamp. Apply a gas pressure adjusted so that the liquid front descends the column at a rate of 7 to 13 mm. per min. (Note 8). Shut off the gas supply at such a time that the pressure in the column is nearly atmospheric when all the sample is adsorbed. Open the reservoir, add a section of about 20 mm. of silica gel, fill the reservoir with alcohol, reconnect to the gas source and readjust the pressure so that the liquid front continues to descend at a rate of 7 to 13 mm. per min.

NOTE 7.—The aromatic content of a sample may be estimated approximately from its refractive index and the refractive index of the first drops of saturates obtained by percolating a small portion of the sample through a short column (150 mm.) of coarse silica gel (28 to 200 mesh) contained in a glass tube 200 mm. in length with an inside diameter of 6 mm. and with a 2-mm. opening at the lower end plugged with glass wool as a retainer for the gel. For the refractive indices of the various aromatics use the values shown in Table III.

NOTE 8.—Normally a pressure of 5 to 10 psi. is sufficient to provide the specified flow rate. If greater pressures are required, it is important to have a transparent protective shield around the column.

(*c*) Take fractions at a rate of 0.3 to 0.7 ml. per min. as follows: When the desired volume has collected in the receiver, close the stopcock, tap it gently to collect any drop suspended from the column outlet, and allow 40 ±5 sec. for draining. Estimate and record the volume to the nearest 0.002 ml. Open the receiver stopcock fully and drain the fraction into the appropriate vial for 40 ±5 sec. Close the stopcock of the receiver and open the column stopcock gently for collection of the next fraction. Cap the vial tightly.

TABLE III.—REFRACTIVE INDICES OF AROMATICS.

Distillation Fraction	Refractive Index, n_D^{25}
Benzene	1.4980
Toluene	1.4940
Whole Gasoline	1.4940
Xylenes	1.4958
Higher	1.494

The volume (5 or 10 ml.) of the saturate or the aromatic fraction taken is governed by the composition of the sample (Table II); do not change receivers during an analysis. After about three fourths of the saturates have been collected, take 1-ml. fractions. The boundary between the saturates and the aromatics may be visible in the silica-gel column and its arrival in the percolate is identified by the appearance of refraction waves or "schlieren." Take 1-ml. fractions until three fractions, in which "schlieren" can no longer be observed, have been collected. Similarly take 1-ml. fractions as the end of the aromatic portion is approached and for 5 ml. beyond this point. The boundary between the aromatics and the alcohol is always visible in the column.

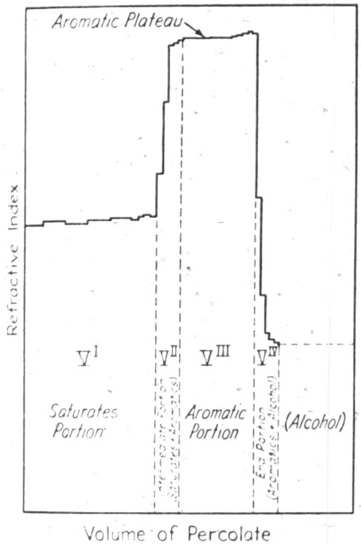


FIG. 3.—Adsorptogram.

(*d*) Measure the refractive indices of the fractions to plus or minus 0.0001 of a unit.

(*e*) Release the pressure, disconnect, and invert the column in its stand, and place beneath it a large pan for collecting the silica-gel slurry. Gently force a flexible $\frac{3}{16}$-in. plastic tube, from which water is issuing, upward through the column to displace the gel, and discard the gel slurry.

Calculations

9. (*a*) Correct all volume measurements about the 5- and 10-ml. marks by multiplying them by the drainage factor, K_5 or K_{10}, obtained as described in Section 5.

(*b*) For the sake of convenience, the refractive indices of the fractions may be plotted against their volumes, forming an adsorptogram as illustrated in Fig. 3, marking off the four major portions of the entire percolate.

(*c*) Calculate the volumes of saturates and of aromatics in the four portions as follows:

(*1*) *Saturates Portion.*—The volume of saturates, V_S^I, is the total volume of this portion, V^I.

(*2*) *Intermediate Portion.*—The volume of the saturates, V_S^{II}, is the sum of the volumes of the saturates in each individual fraction, and is calculated as follows:

$$V_S^{II} = \Sigma \left[\frac{n_A - n_f}{n_A - n_S} \times V_f \right]$$

where:

n_A = refractive index of the aromatic "plateau" in the aromatic portion (Note 9),

n_f = refractive index of an individual fraction,

n^S = refractive index of the last 1-ml. fraction of the saturates portion, and,

V_f = volume of the individual fraction having refractive index n_f.

The volume of aromatics, V_A^{II}, in the intermediate portion is obtained by difference as follows:

$$V_A^{II} = V^{II} - V_S^{II}$$

where:

V^{II} = total volume of the intermediate portion.

NOTE 9.—Select the proper value of the refractive index of the aromatic "plateau" from the following considerations:

(*a*) If a definite portion of the aromatic "plateau" has a refractive index constant within plus or minus 0.0003, use this value.

(*b*) If no portion of the aromatic "plateau" has a refractive index constant to plus or minus 0.0003 but, as is usually the case, adjacent 1-ml. fractions differ by 0.0004 or less, use the refractive index of the first 1-ml. fraction which does not differ by more than 0.0004 from that of the succeeding 1-ml. fraction.

(*c*) If no definite aromatic "plateau" is obtained and it is apparent that no pure aromatic fraction has been obtained (as in the case of low-aromatic content samples) use the values interpolated from Table III.

(*3*) *Aromatic Portion.*—The volume of aromatics, V_A^{III}, is the volume of the portion, V^{III}.

(*4*) *End Portion.*—The volume of aromatics is the sum of the volumes of the aromatics, V_A^{IV}, in each individual fraction, and is calculated as follows:

$$V_A^{IV} = \Sigma \left[\frac{n_f - n_a}{n_A - n_a} \times V_f \right] - C$$

where:

n_A = refractive index of the last 1-ml. fraction of the aromatic portion,

n_f = refractive index of an individual fraction,

n_a = refractive index of the alcohol; use 1.3600 for ethyl alcohol and 1.3755 for isopropyl alcohol (Note 5),

V_f = volume in milliliters of the individual fraction having refractive index n_f, and

C = volume of the gel-alcohol blank, in milliliters (Section 6 (*b*)).

(*d*) Calculate in milliliters, the total volume of saturates, V_S, and the total volume of aromatics, V_A, in the percolate as follows:

$$V_S = V_S^I + V_S^{II}$$

$$V_A = V_A^{II} + V_A^{III} + V_A^{IV}$$

(*e*) Calculate the percentage by volume of aromatics, A_u, in terms of the sum of the unmixed aromatics and saturates as follows:

$$A_u = \frac{A}{A + S} \times 100$$

where:

A = volume in milliliters of aromatics, and

S = volume in milliliters of saturates.

(*f*) Calculate the percentage by volume of aromatics obtainable from the original sample, A_0, as follows:

$$A_0 = \frac{A_u}{100 + \Delta V} \times 100$$

where:

ΔV = percentage change in volume on mixing the separated components (Note 10).

NOTE 10.—The difference between A_u and A_o is greatest at nearly equimolar mixtures and seldom exceeds 0.1 per cent. The largest change occurs in the nearly equimolar mixture of benzene and cyclohexane where the difference between A_u and A_o is about 0.3 per cent. To determine ΔV combine all the saturate fractions (Fig. 3) in one sample container and all aromatic fractions in a second sample container, omitting all fractions which are intermediate in composition. The procedure in the Appendix may be followed.

Precision and Accuracy

10. (*a*) Results should not differ from the mean by more than the following amounts:

Aromatic Content, per cent by volume	**Repeatability,** One Operator and Apparatus	**Reproducibility,** Different Operators and Apparatus
15 to 75.......	0.15	0.2

(*b*) Results should not differ from the true value by more than the following (Note 11):

Aromatic Content, per cent by volume	Accuracy
15 to 75................	±0.2

NOTE 11.—The determined value is more often greater than less than the true value.

APPENDIX

MEASUREMENT OF VOLUME CHANGE ON MIXING LIQUID HYDROCARBONS

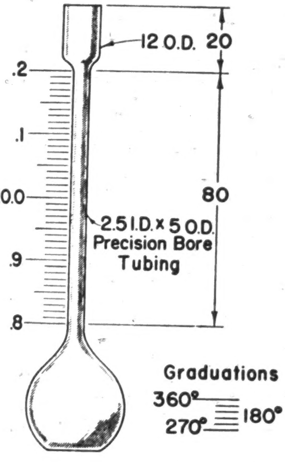
Apparatus:

A1. (*a*) *Volumetric Flask,* 20-ml. capacity, as shown in Fig. 4. The exact volume of the flask is not required, but the relative volumes at the graduation marks must be known.

(*b*) *Pipette Assembly* as illustrated in Fig. 5. The 2.0-ml. pipette need not be calibrated but should have a water-delivery time of 22 sec. or greater.

Procedure:

A2. (*a*) Place the combined aromatic fractions, the combined saturate fractions, a portion of the original sample, and the 20-ml. volumetric flask in a dish or bath filled with water at room temperature, weighting the three containers and the flask to prevent tipping.


12 O.D.
20
.2
.1
20.0
80
2.5 I.D. × 5 O.D. Precision Bore Tubing
.9
.8
Graduations
360°
270°
180°

All dimensions in millimeters.

FIG. 4.—Volumetric Flask.

(*b*) From the silica-gel adsorption analysis, divide the aromatic content of the sample, in per cent, by 10 and round off the quotient to the nearest integer. Denote the result as "*n*."

NOTE 12.—The total number of aromatic portions used in the experiment is the determined value "*n*"; "10-*n*" is the total number of non-aromatic portions used.

(*c*) By means of the pipette arrangement shown in Fig. 5, transfer to the volumetric flask (10-*n*) 2-ml. portions of the combined saturate fractions, followed by (*n*-1) 2-ml. portions of the combined aromatic fractions.

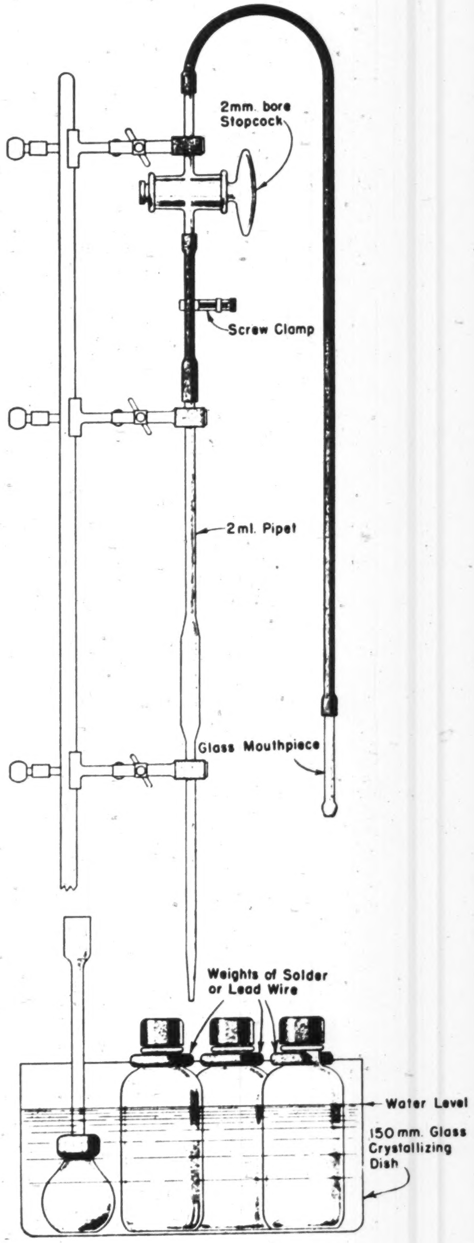

2mm. bore Stopcock
Screw Clamp
2 ml. Pipet
Glass Mouthpiece
Weights of Solder or Lead Wire
Water Level
150 mm. Glass Crystallizing Dish

FIG. 5.—Pipette Assembly.

To transfer the portions from the sample bottle to the flask, draw the material into the pipette to slightly above the graduation mark and close the stopcock. By partially closing the pinch clamp, adjust the miniscus exactly to the mark. Remove any drops of liquid by touching the tip of the pipette lightly with a piece of filter paper held in a nearly vertical position. Touch the tip of the pipette to the inside of the widened portion of the neck of the flask and open the stopcock fully. Replace the flask in the water bath as soon as the flow of liquid from the pipette ceases.

NOTE 13.—It is essential that the temperature of the water bath remain constant within plus or minus 0.3 C. for any series of ten 2-ml. portions transferred.

(*d*) Mix the contents of the flask as completely as possible by shaking with a sideways motion. Bring into the body of the liquid any material in the neck of the flask, by shaking the flask with a short downward motion, or by tilting the flask sideways until the body of the liquid joins with that in the neck, then slowly return the flask to a vertical position. Add the final (*n* th) 2-ml. portion of the combined aromatic fractions. Shake the flask gently with a sideways motion, return to the water bath for 10 min., and finally read the volume to 0.002 ml.

(*e*) Empty the flask, dry, and return to the water bath. Using the same pipette assembly and the same pipetting and mixing procedure as described above, transfer ten 2-ml. portions of the original sample to the flask, equilibrate to temperature for 10 min., and read the volume to 0.002 ml.

NOTE 14.—No significant error is introduced if the "constant" temperature for the second series of ten 2-ml. portions transferred to the flask is different from that of the first series.

NOTE 15.—If the quantity of the original sample is less than 20 ml. a portion of the mixture from the first series of transfers (Paragraph (*c*)) may be removed from the volumetric flask in the manner illustrated in Fig. 6 and a small amount of the original sample added to give a volume slightly in excess of 20 ml.

Calculation:

A3. Calculate the percentage volume change, ΔV, as follows:

$$\Delta V = 5(V_1 - V_2)$$

where:

V_1 = observed total volume of n 2-ml. portions of the combined aromatic fractions and $(10 - n)$ 2-ml. portions of the combined saturate fractions, in milliliters, and

V_2 = observed total volume of ten 2-ml. portions of the original sample.

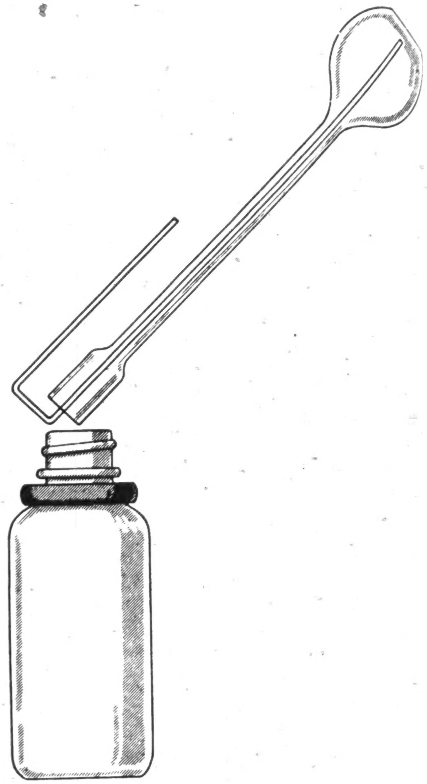


FIG. 6.—Emptying the Flask.

REPORT OF COMMITTEE D-3*

ON

GASEOUS FUELS

Committee D-3 on Gaseous Fuels and its seven subcommittees held a joint meeting in New York, N. Y., on October 23, 1945. A meeting of Subcommittee I was also held on the same date. Advantage was taken of the Spring Group Committee Meetings in Pittsburgh, Pa. for another joint meeting of Committee D-3 and its subgroups on February 26, 1946. Subcommittees II, III, IV, and VI all met on the preceding day. With the lifting of restrictions imposed by war conditions and return to more normal activities, very substantial progress has been made in the program sponsored by Committee D-3. This will be evident from the following accounts of the activities of its individual groups.

New Tentative Method

Committee D-3 recommends that the Method of Test for Calorific Value of Gaseous Fuels by the Water-Flow Calorimeter,[1] developed by Subcommittee III, be accepted for publication as tentative.

This recommendation has been submitted to letter ballot of the committee, which consists of 35 members; 29 members returned their ballots, all of whom voted affirmatively.

Activities of Subcommittees

Subcommittee I on Collection of Gaseous Samples (S. J. Modzikowski, chairman).—Following the reorganization of the work of this group, a second revision of a proposed Tentative Method for Sampling Liquefied Petroleum Gases has been prepared. Attention has also been given to the preparation and acceptance of terms in common use. Preparation of methods for the sampling of manufactured and natural gases is also planned. It is proposed to publish the methods individually as soon as they are completed.

Subcommittee II on Measurement of Gaseous Samples (H. S. Bean, chairman).—Principal attention has been devoted by this group to small wet laboratory meters, and it is now in readiness to proceed with the preparation of material on their calibration and use. It is expected that a tentative draft will be available at the fall meeting of Committee D-3. Consideration has also been given to including reference to dry gas measurement in any standards prepared by the committee and agreement was reached that this should be done.

Subcommittee III on Determination of Calorific Value of Gaseous Fuels (R. S. Jessup, chairman).—Following a great deal of work by a special subgroup, a Tentative Method of Test for Calorific Value of Gaseous Fuels by the Water-Flow Calorimeter was prepared and submitted to all members of the subcommittee. Detailed specifications for a gas calorimeter inlet thermometer, gas calorimeter outlet thermometer, and gas calorimeter flue thermometer for use in the test were prepared in cooperation with the Section on Thermometers, of Committee E-1 on Methods of Testing. At the February meeting the test method and the thermometer specifications were approved with minor revisions. The revised text was later submitted to all members of both Committee D-3 and the subcommittee for simultaneous letter ballot. Approval has since been given by more than a two-thirds majority of both groups.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

[1] See Editorial Note, p. 366.

Subcommittee IV on Determination of Specific Gravity and Density of Gaseous Fuels (E. F. Schmidt, chairman).—Agreement was reached by this group at its February, 1946, meeting on a definition of specific gravity for presentation to all members for approval. Consideration was also given to tolerances for specific gravity instruments and agreement reached to recommend a definite tolerance following a further study of the accuracy of which the various instruments tested by the National Bureau of Standards were capable. It is planned to secure an expression from the committee by letter ballot following the submission of the necessary data.

Subcommittee VI on Determination of Water Vapor Content of Gaseous Fuels (A. W. Gauger, chairman).—An additional investigation was undertaken of a water-vapor detector, the principle of operation of which is based on change in conductivity of a film of phosphoric acid as affected by the absorption of moisture. Equipment loaned by the company developing this detector is now under test. Attention has also been given to a procedure for moisture determination making use of calcium nitride, ammonia being liberated on contact with water vapor.

Subcommittee VII on Complete Analysis or Chemical Composition of Gaseous Fuels (Martin Shepherd, chairman).—A research paper covering results of analysis of the first cooperative sample distributed by the National Bureau of Standards is planned for early release. A report on the second sample is in readiness for committee review. Samples for mass spectrometer analysis have been prepared and distributed. A cross check is planned between the resulting analyses and those obtained by chemical methods. Assistance of a number of selected laboratories will be utilized in securing this information.

The election of officers for the ensuing term of two years resulted in the election of the following:

Chairman, A. W. Gauger.
Vice-Chairman, E. F. Schmidt.
Secretary, R. M. Conner.

In addition, A. C. Fieldner (alternate, G. W. Jones), J. V. Freeman, E. O. Mattocks, and Martin Shepherd were elected members of the Advisory Committee.

This report has been submitted to letter ballot of the committee, which consists of 35 members; 32 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. W. GAUGER,
Chairman.

R. M. CONNER,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-3 presented to the Society through the Administrative Committee on Standards the proposed Tentative Method of Test for Calorific Value of Gaseous Fuels by the Water-Flow Calorimeter. The new tentative method was accepted by the Standards Committee on September 9, 1946, and appears in the 1946 Book of A.S.T.M. Standards, Part III-A, under the designation D 900 – 46 T.

REPORT OF COMMITTEE D-4*

ON

ROAD AND PAVING MATERIALS

Because of war conditions, Committee D-4 on Road and Paving Materials was unable to hold any meeting or present an annual report to the Society during 1945. It did, however, meet at Pittsburgh, Pa., on February 28, 1946, at which meeting action was taken on the recommendations in this report.

I. New Tentative Method

Committee D-4 recommends that the Tentative Method of Test for Sulfonation Index of Road Tars, appended hereto,[1] be accepted for publication as tentative. This method of test is identical in substance with Method T 108 of the American Association of State Highway Officials, as revised by that organization in 1945.

II. Adoption of Tentatives as Standard

The committee recommends that the following three tentatives, with revisions in one method as indicated, be approved for reference to letter ballot of the Society for adoption as standard:

Tentative Specifications for Cut-Back Asphalt (*Rapid Curing Type*) (**D 597 - 40 T**),[2] without revision,

Tentative Specifications for Cut-Back Asphalt (*Medium Curing Type*) (**D 598 - 40 T**),[2] without revision, and

Tentative Method of Test for Abrasion of Gravel by Use of the Deval Machine (**D 289 - 42 T**),[2] revised as follows:

Title.—Delete the word "Gravel" and substitute "Graded Coarse Aggregate."

Section 1.—Change the first sentence to read as follows by the addition of the italicized words: "This method covers the procedure for testing uncrushed or crushed gravel, *crushed stone, or crushed blast-furnace slag* for resistance to abrasion in the Deval testing machine with an abrasive charge."

Section 4.—Delete the word "gravel" and substitute "coarse aggregate."

Section 5 (*a*).—In the first two sentences delete the word "gravel" and substitute "coarse aggregate." In the table, opposite grading D, change "1/4 in." to read "No. 4."

Section 5 (*c*).—Delete the word "gravel" and substitute "coarse aggregate."

Section 7 (*a*).—Change to read as follows by the addition of the italicized words and the omission of those in brackets: "7. (*a*) [Uncrushed gravel.—In the case of uncrushed gravel] *Percentage of wear.*—The loss by abrasion shall be considered as the difference between the original weight of the test sample and the weight of the material retained on the No. 12 (1680-micron) sieve, expressed as a percentage of the original weight of the test sample."

Method D 289 was withdrawn from the standards of the Society in 1940, on the assumption that it had been largely supplanted by the Standard Method of

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

[1] This method was accepted as tentative by the Society and appears in the 1946 Book of A.S.T.M. Standards, Part II.

[2] 1944 Book of A.S.T.M. Standards, Part II.

Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131). However, protests against its withdrawal were immediately received and it developed that a considerable number of agencies continued to specify the method. Accordingly, it was reinstated without change in 1942. Because it has been quite commonly used for tests of coarse aggregates other than gravel, its scope has been enlarged by the above proposed revision, which is recommended by Committee D-4, subject to concurrence of Committee C-9.[3]

III. REVISION OF STANDARDS, IMMEDIATE ADOPTION

For reasons later set forth, Committee D-4 recommends that the following revisions of standards be immediately incorporated in these standards. The committee accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society:

Standard Specifications for Slow-Setting Emulsified Asphalt (for Fine Aggregate Mixes) (**D 631 – 43**):

Section 2 (*b*).—Change the second requirement to read as follows by the omission of the words and figures in brackets: "Residue [by evaporation at 163 C. (325 F.) or] by distillation. . . ."

Section 2 (*c*).—Change the first sentence to read as follows by the omission of the words in brackets: "The residue obtained from [evaporation or] distillation shall conform to the following requirements:"

These changes are recommended to correct an error as no test for residue by evaporation has as yet been included in Standard Methods of Testing Emulsified Asphalts (D 244).

Standard Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (**C 131 – 44**).—The following revision is submitted by Committee D-4, subject to concurrence of Committee C-9.[3]

Section 5.—Add the following sentence after the first sentence in this section: "The machine shall be so driven and so counterbalanced as to maintain a substantially uniform peripheral speed."

Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates (**C 136 – 39**).—Committee D-4 recommends[3] the following revision:

Section 3 (*a*).—Change to read as follows by the addition of the italicized words and the omission of those in brackets:

> 3(*a*).—Samples for sieve analysis shall be obtained [by quartering or by use of a sampler, from a representative sample selected from the material to be tested] *from the materials to be tested by the use of a sample splitter or by the method of quartering. Fine aggregate sampled by the quartering method shall be thoroughly mixed and in a moist condition. The sample for test shall be approximately of the weight desired and shall be the end result of the sampling method. The selection of samples of an exact predetermined weight shall not be attempted.*

Section 3 (*b*).—Change the first sentence to read as follows by the addition of the italicized words and figures and the omission of the words in brackets: "(*b*) Samples of fine aggregate for sieve analysis shall weigh after drying [not less than] *approximately* the amount indicated in the following table:"

After the table, add the following sentence and footnote:

> In no case, however, shall the fraction retained on any sieve at the completion of the sieving operation weigh more than 4 g. per sq. in. of sieving surface.[a]
>
> [a] This amounts to 200 g. for the usual 8-in. diameter sieve. The amount of material retained on the critical sieve may be regulated by: (*1*) the introduction of a sieve having larger openings than in the critical sieve or, (*2*) by the proper selection of the size of the sample.

Standard Definitions of Terms Relating to Materials for Roads and Pavements

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[3] See p. 317

(**D 8 – 33**).—The present definition for asphalt cements contains an upper penetration limit of 250, which was sufficiently high to cover products commonly designated as asphalt cements at the time the definition was adopted. Since then, Federal Specification SS-A-706B and specifications of a number of state highway departments have called for asphalt cements with a penetration as high as 300. Committee D-4 therefore recommends that the definition be changed by substituting an upper penetration limit of "300" for the present limit of "250."

IV. REVISION OF STANDARDS AND REVERSION TO TENTATIVE

Committee D-4 recommends that the following standards be revised and reverted to tentative:

Standard Specifications for Calcium Chloride (**D 98 – 34**):

Section 2.—Change the second and third requirements to read as follows by the addition of the italicized words and the omission of the word in brackets:

Total magnesium [chloride] *as* $MgCl_2$ max., per cent	0.5
Total alkali chlorides *calculated as NaCl*, max., per cent	2.0

Standard Methods of Chemical Analysis of Calcium Chloride (**D 345 – 34**).—Revise to conform with the tentative revisions submitted August, 1941,[2] except that in the new Section 3, substitute "3/8 in." for "3/4 in." as a sieve designation. In addition the following changes are recommended:

Section 2.—Renumber as Section 4 and change the first four sentences of Paragraph (*a*) to read as follows by the addition of the italicized words and figures and the omission of those in brackets:

> Weigh accurately *in a weigh bottle* a sample of approximately [5] *50* g. (Note 1) and dissolve in *distilled* water. Add sufficient HCl to clear, and then make up to [a volume of] 500 ml. *in a volumetric flask and mix thoroughly. Pipette a 50-ml. aliquot into another 500-ml. volumetric flask, make to volume and mix thoroughly.* Pipette a [25.00] *25* ml. aliquot and add enough diluted NH_4OH to make slightly alkaline [Methyl orange or red may be used as indicator] *to methyl orange or methyl red.*

Explanatory Notes.—Change Note 1 to read as follows:

> NOTE 1.—Instead of weighing out separate samples as indicated in Sections 4, 5, and 6, a 50-g. sample may be dissolved and diluted to 500 ml. in a volumetric flask with distilled water. For the procedure in Section 4, pipette a 50-ml. aliquot into another 500-ml. volumetric flask and dilute to volume. From this second flask, containing 5 g. of original sample, pipette a 25-ml. aliquot as indicated in Section 4 and continue the procedure for total calcium as outlined. For the procedure in Section 5, measure from a burette a 100-ml. aliquot which will represent a 10-g. sample for alkalinity and magnesium compounds. For the procedure in Section 6, measure 80 ml. of solution from a burette, which will represent 8 g. of sample for the alkali chlorides. This handling will render accurate distribution of the insoluble $Mg(OH)_2$ and $CaCO_3$ present more difficult than using direct weighed samples. However, if each volumetric flask is well mixed before each portion is removed, fairly accurate results can be expected. Instead of weighing accurately a 10.00-g. sample, a sample of approximately 10 g. may be used if desired, in which case the percentage of $Ca(OH)_2$ may be calculated as follows:
>
> $$Ca(OH)_2\text{, per cent} = \frac{\text{ml. of } 0.1\ N\ \text{HCl} \times 0.0037 \times 100}{\text{wt. of sample in g.}}$$

V. REVISION OF TENTATIVES

Committee D-4 recommends that the following two tentative methods be revised as follows:

Tentative Methods of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials (**D 75 – 42 T**):

Section 14 (*a*).—Change the third sentence from its present form: namely,

> Separate samples shall be taken from the top, middle, and bottom of each unit of the shipment.

to read as follows:

> Separate samples shall be taken from three or more points of each unit of the shipment, each sample representing, as nearly as possible, the average of the unit as indicated by careful observation.

Add the following note at the end of this section:

> NOTE.—Samples from stockpiles should be taken at or near the top of the pile, at or near the base of the pile, and at an intermediate point. A board shoved into the pile just above the point of sampling will aid in preventing further segregation during sampling. Samples from railroad cars should be taken from three or more trenches dug across the car at points which appear on the surface to be representative of the material. The bottom of the trench should be at least 1 ft. below the surface of the aggregate at the sides of the car and approximately 1 ft. wide at the bottom. The bottom of the trench should be practically level. Equal portions should be taken at nine equally spaced points along the bottom of the trench by pushing a shovel downward into the material and not by scraping horizontally. Two of the nine points should be directly against the sides of the car. Fine aggregate may be sampled from either stockpiles, trucks, or railroad cars by the same procedure or by means of a sampling tube approximately 1 ¼ in. in diameter by 6 ft. which, with a little practice, will be found to hold damp sand forced into it when inserted into the fine aggregate to be sampled. Five to eight insertions of the tube into the unit to be sampled will furnish approximately 10 lb. of fine aggregate.

Tentative Methods of Sampling Bituminous Materials (**D 140 - 41 T**).—These methods are under the joint jurisdiction of Committee D-4 and Committee D-8 on Bituminous Waterproofing and Roofing Materials. Committee D-8 concurs in the following proposed revision:

Section 8 (*a*).—Change the third and fourth sentences to read as follows by the addition of the italicized words and the omission of the words and figure in brackets:

> This pipe shall be provided with a plug cock and shall discharge into a [receiving drum of 50-gal. capacity] *sample receiver.* The plug cock shall be so adjusted that there is a steady continuous flow of bituminous material through it and [shall be] so regulated that the *sample* receiver will fill *to the required amount* in the same time that is required to make the entire pumping.

Section 13.—Change this section to read as follows by the addition of the italicized words and figures:

> 13. Solid bituminous materials in crushed fragments or powder, such as gilsonite, grahamite, etc., shall be sampled in accordance with the Standard Method of Sampling Coal for Analysis (A.S.T.M. Designation: D 21), *except that the gross sample may be less than 1000 lb. but not less than 50 lb. when knowledge of the uniformity of the solid bituminous material in the shipment warrants.*

VI. STANDARDS AND TENTATIVES CONTINUED WITHOUT REVISION

Committee D-4 recommends continuance in their present status of the following standards and tentatives most of which have been recently reviewed by subcommittees responsible for their development:

Standard Specifications for:

Coal-Tar Pitch for Stone Block Filler (D 112 - 30),
Medium-Setting Emulsified Asphalt (for Coarse Aggregate Plant Mixes) (D 397 - 39),
Medium-Setting Emulsified Asphalt (for Retread and Coarse Aggregate Mixes) (D 398 - 39)
Medium-Setting Emulsified Asphalt (Heavy Premix—Summer Grade) (D 399 - 39)
Quick-Setting Emulsified Asphalt (for Penetration and Surface Treatment) (D 401 - 40)
Sand for Sheet Asphalt and Bituminous Concrete Pavements (D 162 - 29),
Mineral Filler for Sheet Asphalt and Bituminous Concrete Pavements (D 242 - 39),
Asphalt Plank (D 517 - 40),
Materials for Cement Grout Filler for Brick and Stone Block Pavements (D 57 - 20),
Materials for Sand-Cement Bed for Brick and Block Pavements (D 58 - 37),
Granite Block for Pavements (D 59 - 39),
Recut Granite Block for Pavements (D 131 - 39), and
Granite Block for Durax Pavements (D 132 - 39).

Standard Methods of Test for:

Distillation of Tar Products Suitable for Road Treatment (D 20 - 30), Distillation of Cut-Back Asphaltic Products (D 402 – 36),

Softening Point of Bituminous Materials (Ring-and-Ball Method) (D 36 – 26),

Softening Point of Tar Products (Cube-in-Water Method) (D 61 – 38),

Float Test of Bituminous Materials (D 139 – 27),

Amount of Material Finer than No. 200 Sieve in Aggregates (C 117 – 37),

Abrasion of Rock by Use of the Deval Machine (D 2 – 33),

Toughness of Rock (D 3 – 18),

Water in Petroleum Products and Other Bituminous Materials (D 95 – 40),

Penetration of Bituminous Materials (D 5 – 25),

Specific Gravity of Road Oils, Road Tars, Asphalt Cements and Soft Tar Pitches (D 70 – 27),

Specific Gravity of Asphalts and Tar Pitches Sufficiently Solid to be Handled in Fragments (D 71 – 27), and

Standard Recommended Practice for Bituminous Paving Plant Inspection (D 290 – 39).

Tentative Specifications for:

Tar (D 490 – 43 T), with the following editorial revision to refer to the new tentative method of test recommended in this report:

Section 3.—In the first sentence delete reference to Paragraph (*h*). In Paragraph (*h*) add a reference to the proposed A.S.T.M. Tentative Method of Test for Sulfonation Index of Road Tars.[1]

Crushed Stone, Crushed Slag, and Gravel for Bituminous Concrete Base and Surface Courses of Pavements (D 692 – 42 T),

Materials for Stabilized Base Course (D 556 – 40 T),

Materials for Stabilized Surface Course (D 557 – 40 T), and

Standard Sizes of Coarse Aggregate for Highway Construction (D 448 – 42 T).

Tentative Methods of Test for:

Hot Extraction of Asphaltic Materials and Recovery of Bitumen by the Modified Abson Procedure (D 762 – 44 T),

Loss on Heating of Oil and Asphaltic Compounds (D 6 – 39 T), and

Moisture-Density Relations of Soils (D 698 – 42 T).

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 125 members; 93 members returned their ballots with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVE METHOD			
Test for Sulfonation Index of Road Tars	42	1	50
II. ADOPTION OF TENTATIVES AS STANDARD			
Spec. for Cut-Back Asphalt (Rapid Curing Type) (D 597 – 40 T)	77	3	13
Spec. for Cut-Back Asphalt (Medium Curing Type) (D 598 – 40 T)	79	2	12
Test for Abrasion of Gravel by Use of the Deval Machine (D 289 – 42 T), as revised	68	0	25
III. REVISION OF STANDARDS, IMMEDIATE ADOPTION			
Spec. for Slow-Setting Emulsified Asphalt (D 631 – 43)	70	1	22
Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 – 44)	72	0	21
Test for Sieve Analysis of Fine and Coarse Aggregates (C 136 – 39)	80	0	13
Definitions of Terms Relating to Materials for Roads and Pavements (D 8 – 13)	86	1	6
IV. REVISION OF STANDARDS AND REVERSION TO TENTATIVE			
Spec. for Calcium Chloride (D 98 – 34)	50	0	43
Methods of Chemical Analysis of Calcium Chloride (D 345 – 34)	46	0	47
V. REVISION OF TENTATIVES			
Methods of Sampling Stone, Slag, Gravel, Sand and Stone Block for Use as Highway Materials (D 75 – 42 T)	76	0	17
Methods of Sampling Bituminous Materials (D 140 – 41 T)	86	0	7

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, W. J. Emmons.

First Vice-Chairman, F. H. Bauman.

Second Vice-Chairman, A. T. Goldbeck.

Third Vice-Chairman, E. W. Klinger.

Secretary, B. A. Anderton.

This report has been submitted to letter ballot of the committee, which consists of 125 members; 93 members returned their ballots, of whom 87 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

SHREVE CLARK,
Chairman.

PRÉVOST HUBBARD,
Secretary.

REPORT OF COMMITTEE D-5*

ON

COAL AND COKE

Committee D-5 on Coal and Coke held a meeting in Chicago, Ill., on February 26, 1946; another meeting was held at the Annual Meeting of the Society in June, 1946, in Buffalo, N. Y. Meetings of a number of subcommittees were also held during the year as recorded later in this report.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, A. C. Fieldner.
Vice-Chairman, D. H. Davis.
Secretary, W. A. Selvig.

The committee sustained a great loss in the death of O. O. Malleis, Vice-Chairman. Mr. Malleis joined the Society in 1916, and was one of the most active workers on the committee. G. H. Nowlin, Jr., resigned during the year and W. M. Berthoff joined the committee. The present membership of Committee D-5 is 54, of whom 14 are classified as producers, 18 as consumers, and 22 as general interest members.

A new subcommittee has been created designated as Subcommittee XX on Sampling and Fineness Test of Pulverized Coal, under the chairmanship of J. B. Romer. This subcommittee is to revise and bring up to date the present Standard Method of Sampling and Fineness Test of Powdered Coal (D 197 – 30) which has not been changed since 1930. The subcommittee will cooperate in this work with a Power Test Code Committee of The American Society of Mechanical Engineers.

I. Recommendations Affecting Standards

Committee D-5 is recommending for adoption as standard two tentative methods and a tentative revision of one standard as listed in Table I.

The Tentative Method of Sampling Coals Classed According to Ash Content (D 492 – 43 T) is recommended for adoption as standard with a minor revision, as follows:

Section 4 (*a*).—Change the second sentence to read as follows by the addition of the italicized word and the omission of the word in brackets; also by the addition of the footnote in italics:

> This procedure is *intended* [designed] for an accuracy such that if a large number of samples were taken from a single lot of coal, the test results in 95 out of 100 cases would fall within plus or minus 10 per cent of the average ash content of these samples.[a]
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> ---
>
> [a] *It is recognized that this accuracy specification may not be obtained for all size groups and ranges of ash shown in Table II. Further sampling investigations are in progress to obtain additional data and may result in revision of the accuracy specification.*

The Tentative Method of Test for Free-Swelling Index of Coal (D 720 – 43 T) is recommended for adoption as standard without revision.

The tentative revision[1] of Standard D 271 being recommended for adoption as standard is a change in the requirements for maximum permissible difference between two or more determinations of volatile matter in coal and coke.

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* **Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.**

[1] **1945 Supplement to Book of A.S.T.M. Standards, Part III, p. 485.**

These recommendations were approved at a meeting of the committee held June 27, 1946, in Buffalo, N. Y., and have been submitted to letter ballot of the committee which consists of 54 members, the results of which are shown in Table I.

II. TENTATIVE METHODS CONTINUED AS TENTATIVE

The committee is recommending that the following tentative methods, which have stood for two years or longer without revision, be continued as tentative since revisions may be desirable before they are adopted as standard:

TABLE I. ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. ADOPTION OF TENTATIVES AS STANDARD			
Methods of Sampling Coals Classed According to Ash Content (D 492 - 43 T), as revised	39	1	3
Test for Free-Swelling Index of Coal (D 720 - 43 T)	38	1	4
II. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD			
Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 - 44)	41	0	2

Method of Test for Grindability of Coal by the Ball-Mill Method (D 408 - 37 T),

Method of Test for Grindability of Coal by the Hardgrove-Machine Method (D 409 - 37 T), and

Method of Drop Shatter Test for Coal (D 440 - 37 T),

III. STANDARDS SIX YEARS WITHOUT REVISION

The following standards which have been published for six years or longer without revision are to be continued as such as no revisions are recommended at this time:

Standard Specifications for:

Gas and Coking Coals (D 166 - 24),
Classification of Coals by Rank (D 388 - 38),
Coals by Grade (D 389 - 37),

Standard Methods of Tests for:

Drop Shatter Test for Coke (D 141 - 23),
Volume of Cell Space of Lump Coke (D 167 - 24),
Sampling and Fineness Test of Powdered Coal (D 197 - 30),
Cubic Foot Weight of Crushed Bituminous Coal D 291 - 29),
Cubic Foot Weight of Coke (D 292 - 29),
Sieve Analysis of Coke (D 293 - 29),
Tumbler Test for Coke. (D 294 - 29),
Size of Anthracite (D 310 - 34),
Sieve Analysis of Crushed Bituminous Coal (D 311 - 30),
Sampling Coke for Analysis. (D 346 - 35),
Screen Analysis of Coal (D 410 - 38),

Standard Definitions of:

Terms Relating to Coal and Coke (D 121 - 30), and
Commercial Varieties of Bituminous and Subbituminous Coal (D 493 - 39).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Methods of Testing (W. A. Selvig, chairman) has recommended for adoption as standard the present tentative revision of Section 43 of the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 - 44) which revision pertains to tolerances in duplicate determination of volatile matter.

Subcommittee II on Nomenclature and Definitions (A. W. Gauger, chairman).—This subcommittee held a meeting on June 27, 1946, in Buffalo, N. Y. It has under consideration the formulation of definitions pertaining to moisture in coal such as total moisture, inherent moisture, surface moisture, and free moisture. Other terms that probably require clarification are "coking" and "caking" as used to describe properties of coal in the carbonization industry and in combustion processes.

Subcommittee VII on Pulverizing Characteristics of Coal (John Van Brunt, chairman) held a meeting on June 28, 1946, in Buffalo, N. Y. The subcommittee has made plans to conduct coöperative studies on an air-flow method for estimation of surface of pulverized coal which, if practical, will shorten considerably the procedures given in the present tentative methods for determination of grindability of coal which now require rather time-consuming sieving methods.

Subcommittee XIII on Coal Sampling (H. F. Hebley, chairman).—At a meeting held in Buffalo, N. Y., on June 27, 1946, it was agreed that the present Tentative Methods of Sampling Coals Classed According to Ash Content (D 492 – 43 T), with some minor revisions, should be submitted to Committee D-5 for acceptance and recommendation to the Society for adoption as standard.

The Special Section on Sampling Coal for Volatile Matter Determination in Connective with City Smoke Ordinances (W. D. Langtry, chairman) is preparing a paper covering a proposed method of sampling, together with supporting data, for publication in the A.S.T.M. BULLETIN.

Subcommittee XV on Plasticity and Swelling of Coal (C. C. Russell, chairman).—At a meeting held in Chicago, Ill., on February 26, 1946, it was agreed to recommend to Committee D-5 that the Tentative Method of Test for Free-Swelling Index of Coal (D 720 – 43 T) be adopted as standard.

The subcommittee has recommended that the various methods of test for expansion and plastic properties of coals as published in the Appendix of the 1943 Report of Committee D-5,[2] together with that proposed by V. J. Altieri, and given in the discussion of the report, be published for information in the next issue of the compilation "A.S.T.M. Standards on Coal and Coke."

Subcommittee XVI on Ignitibility of Coal and Coke (R. A. Sherman, chairman).—This subcommittee has conducted considerable research on an ignitibility method described in the paper "A Laboratory Test for the Ignitibility of Coal," by Ralph A. Sherman, J. M. Pilcher, and H. N. Ostborg.[3] The subcommittee is of the opinion that the method is still in the research stage and that more work needs to be done before it can be proposed as a tentative method.

Subcommittee XVIII on Coal Classification (H. J. Rose, chairman).—During the past several years this subcommittee has not been active, since coal classification problems did not seem urgent enough to require action under wartime conditions. There has been, however, a realization of the need for a rapid quantitative method of measuring the reflectivity or "brightness" of coal as related to coals of various ranks and types, and the development of some convenient method of quantitatively estimating the percentages of various petrologic components in coal. At a meeting of a subgroup of the subcommittee held in Pittsburgh, Pa., on December 27, 1945, plans were formulated for conducting such research.

This report has been submitted to letter ballot of the committee, which consists of 54 members; 42 members returned their ballots, all of whom have voted affirmatively.

Respectively submitted on behalf of the committee,

A. C. FIELDNER,
Chairman.

W. A. SELVIG,
Secretary.

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[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 301 (1943).

[3] ASTM BULLETIN, No. 112, October, 1941, p. 23.

REPORT OF COMMITTEE D-6*

ON

PAPER AND PAPER PRODUCTS

Committee D-6 on Paper and Paper Products held no meeting during the year due to the restrictive nature of the 1945 Annual Meeting of the Society.

Cooperative relations are being continued between Committee D-6 and other A.S.T.M. committees, as well as with other standardizing bodies.

At the present time, Committee D-6 consists of 77 members, of whom 63 are voting members; 25 are classified as producers, 25 as consumers, 22 as general interest, and 5 as consulting members.

I. Revisions of Tentatives

The committee recommends that the following three methods be revised as indicated and continued as tentative:

Resistance of Paper to Passage of Air (**D 726 - 43 T**), revised as appended hereto[1]

Ply Adhesion of Paper or Vulcanized Fibre (**D 825 - 45 T**), revised as appended hereto[1]

Tentative Method of Qualitative Examination of Mineral Filler and Mineral Coating of Paper (**D 686 - 43 T**), revised as follows:

Section 2 (a).—Add the following sentence parenthetically at the end of this paragraph: "(A 30 per cent solution of H_2O_2 may be used in proportionately lesser quantity)."

Section 2 (q).—Add the following as a new Paragraph (*q*), relettering the present Paragraph (*q*) and subsequent paragraphs accordingly: "(*q*) *Microcosmic Salt Solution*.—Dissolve 50 g. of $NaNH_4HPO_4$ in water and dilute to 1 liter."

Section 4 (b).—Add the following note under item (*3*):

Note.—So far as is known, mixtures of sulfides and sulfites are not used in loading or coating paper. To test for CO_2 in the presence of sulfites, add to the contents of the beaker a weak solution of iodine (about 0.1 *N*) until the liquid becomes yellow. Sulfites are oxidized to sulfates. Test the evolved gas for CO_2 in the usual manner with lime water.

Section 4 (e).—To correct two typographical errors in this section, make the following corrections:

At the end of the first sentence, change "of sulfite" to read "or sulfite."

In the last sentence in parentheses of item (*2*) change to read as follows: "(Sulfates may be reduced to sulfides, or sulfides oxidized to sulfates, depending on ignition conditions.)"

Section 4.—Add the following as a new Paragraph (*h*):

(*h*) Place the unfolded filter paper containing the acid-insoluble residue (Paragraph (*f*)) in a platinum crucible (Note 1). Dry and ignite with free access of air until all organic matter is removed. Add 1 to 2 g. of Na_2CO_3 and fuse until a clear melt is obtained or all reaction has ceased. Decompose the melt in 25 ml. of hot water, heat to boiling, and wash thoroughly with water. Reserve the filtrate and first two washings for analysis of the water soluble portion according to (*1*) below. Wash the insoluble portion from the filter paper back into the original beaker with a stream of water, place the beaker under the funnel and pour 10 ml. of hot reagent grade HCl (sp. gr. 1.18) through the filter paper. Reserve the contents of the beaker for analysis of the water insoluble portion according to (*2*) below.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] These revised methods were accepted by the Society and appear in the 1946 Book of A.S.T.M. Standards, Part III-B.

(*1*) Acidify the water soluble portion with HCl, evaporate to dryness, and bake at 100 to 105 C. for 1 hr. Moisten the residue with reagent grade HCl (sp. gr. 1.18), let stand for a few minutes, add 5 to 10 ml. of diluted HCl and warm. The presence of silicate is indicated by a light, flocculent insoluble residue in the solution, best observed against a dark background. Filter and add $BaCl_2$ solution to a portion of the filtrate. A precipitate indicates sulfates. To another portion of the filtrate add NH_4OH. A precipitate indicates aluminum.

(*2*) Warm the acid solution of the water-insoluble portion, add 50 ml. of water, boil and add a very slight excess of NH_4OH to precipitate aluminum (and iron). Filter and wash, reserving the precipitate for analysis according to (*3*) below. Add 5 ml. of dilute H_2SO_4 (5:95). A precipitate indicates barium. Boil and filter. Add a slight excess of NH_4OH to the filtrate followed by ammonium oxalate solution to insure the absence of calcium, filtering if any precipitate results. Cool the solution and add 5 ml. of NH_3OH and an excess of microcosmic salt solution. Stir well. If a precipitate forms within 15 min., the presence of magnesium is indicated.

(*3*) Wash the insoluble precipitate obtained by the NH_4OH treatment in (*2*) above with water and transfer it to the original beaker with a stream of water (Note 2). Add 5 ml. of HCl and warm. If solution is not complete, add 3 ml. of reagent grade H_2SO_4 (sp. gr. 1.84) and heat under a hood until SO_3 fumes are formed. Cool and dilute to 35 ml. Any barium sulfate not decomposed by the Na_2CO_3 fusion will be insoluble and should be removed by filtration. Make solution neutral with NaOH solution plus an additional volume equal to that of the neutralized solution. Boil, cool and filter. An insoluble precipitate indicates titanium. Neutralize the filtrate with HCl (sp. gr. 1.12), boil and add a slight excess of NH_4OH. A precipitate indicates aluminum.

NOTE 1.—The procedure described in Paragraph (*h*) will serve as a check on the examination as outlined in Paragraphs (*a*) to (*g*).

NOTE 2.—The procedure described in (*3*) is necessary only if both titanium and HCl-insoluble aluminum compounds are present, and it is desired to estimate the relative amounts of each.

New Section.—Add the following as a new Section 6:

6. *Report.*—Report all cations and anions and radicles found present indicating the relative amounts of each, such as trace, slight amount, considerable, large amount, etc. The report should also interpret the results of the analysis in terms of the fillers or mineral coating materials indicated to be present.

II. ADOPTION OF TENTATIVES AS STANDARDS

The committee recommends that the following six tentative methods be approved without change for reference to letter ballot of the Society for adoption as standard:

Tentative Methods of Test for:[2]

Bursting Strength of Paper (D 774 - 44 T),
Effect of Heating on Folding Endurance of Paper (D 776 - 44 T),
Flammability of Treated Paper and Paperboard (D 777 - 44 T),
Hydrogen Ion Concentration (pH) of Paper Extract (D 778 - 44 T),
Time of Penetration by Water of Sized Paper and Paper Products (Dry Indicator Method) (D 779 - 44 T), and
Printing Ink Permeation of Paper (Castor Oil Test) (D 780 - 44 T).

III. REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revisions in the Standard Method of Test for Resin in Paper (D 549 - 41)[2] and accordingly asks for a nine-tenths affirmative vote at the annual meeting in order that these modifications may be referred to letter ballot of the Society:

Title.—Add "and Paperboard" after "Paper."

Section 1.—Change to read as follows:

1. This method covers the procedures for qualitative and quantitative determination of rosin (colophony) in paper and paperboard.[a] The qualitative tests indicate the presence or

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[2] 1944 Book of A.S.T.M. Standards, Part III.

absence of resin from rosin or rosin soap added as a sizing agent. The quantitative tests give the total rosin which consists of the natural resins in the pulp from which the paper is made plus any rosin added as sizing. Such procedures are not applicable to the determination of synthetic resins (such as the melamine and urea-formaldehyde resins used to impart wet strength properties to paper). The method is limited to rosin, which is a natural vegetable resin, the residue left after distilling off the turpentine from the gum of pine or from the solvent extraction of pine stumps, knots and top wood with subsequent separation from the extraction solvent, turpentine or pine oil, by distillation.

Sections 2, 6, and 7.—In these sections and also in the note under the heading "Quantitative Determination," change the word "resin" to read "rosin" wherever it appears.

Section 2.—In the last line, change "resin in papers" to read "rosin or rosin soap added as a sizing material."

Section 6 (c).—At the end of this paragraph, add the following note: "NOTE.—The time of extraction specified may not be sufficient for certain heavy types of paperboard, such as container board, pressboard, binders board and pasted board, and for such paperboards the extraction procedure may not yield strictly quantitative results."

Section 6 (d).—At the end of this paragraph, add the following note: "NOTE.—There is a possibility that there may be present materials other than rosin which might be extracted and therefore would be reported as rosin."

IV. TENTATIVE METHODS CONTINUED AS TENTATIVE

The remaining tentative methods under the jurisdiction of Committee D-6, although requiring no revision, are recommended for continuation as tentative.

The recommendations appearing in this report have been submitted to letter ballot of the committee, which consists of 63 voting members; 55 members returned their ballots, with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Paper Testing Methods (W. R. Willets, chairman) has held two meetings during the year. The subcommittee has been active in developing new methods of test and has also continued its critical study of various methods of test of paper and paper products that have been adopted by other agencies, for example, those of the Technical Association of the Pulp and Paper Industry. The seven tentative methods being recommended for adoption as standards are also under the jurisdiction of this subcommittee.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. REVISIONS OF TENTATIVES			
Qualitative Examination of Mineral Filler and Mineral Coating of Paper (D 686 - 43 T)	43	0	12
Test for Resistance of Paper to Passage of Air (D 726 - 43 T)	46	0	9
Test for Ply Adhesion of Paper or Vulcanized Fibre (D 825 - 45 T)	36	1	18
II. ADOPTION OF TENTATIVES AS STANDARD			
Test for Bursting Strength of Paper (D 774 - 44 T)	47	0	8
Test for Effect of Heating on Folding Endurance of Paper (D 776 - 44 T)	43	0	12
Test for Flammability of Treated Paper and Paperboard (D 777 - 44 T)	42	0	13
Test for Hydrogen Ion Concentration (pH) of Paper Extracts (D 778 - 44 T)	45	1	9
Test for Time of Penetration by Water of Sized Paper and Paper Products (Dry Indicator Method) (D 779 - 44 T)	44	1	10
Test for Printing Ink Permeation of Paper (Castor Oil Test) (D 780 - 44 T)	41	1	13
III. REVISION OF STANDARD, IMMEDIATE ADOPTION			
Test for Resin in Paper (D 549 - 41)	45	0	10

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[a] For further information, see "Paper and Paperboard, Characteristics, Nomenclature and Significance of Tests," p. 78, Am. Soc. Testing Mats., (1944). (Monograph issued as separate publication.)

Methods on the following subjects are being given consideration by Subcommittee I:

- Elongation,
- Analysis for sulfates and chlorides,
- Absorptiveness of paper to paraffin wax,
- Siliceous grit content,
- Retentiveness of filter paper,
- Filtration time of filter paper,
- Ash of filter paper,
- Lint,
- Creasing,
- Stiffness,
- Titanium dioxide content,
- Gloss, and
- Fiber identification.

In addition to the above subjects, studies are being made which may lead to proposed revisions in the Standard Methods of Test for Basis Weight of Paper and Paper Products (D 646 – 44), and in the Tentative Methods of Test for Water Vapor Permeability of Paper and Paperboard (D 783 – 44 T), and for Water Vapor Permeability of Paper and other Sheet Materials at Elevated Temperature and Humidity (D 830 – 45 T).

Subcommittee I also has developed a method of test for bacteriological examination of paper and paperboard. This is still under consideration and is, therefore, not being submitted for publication as tentative, but is included as an Appendix to this report for further information and criticism.

Subcommittee II on Significance of Test Methods (C. C. Heritage, chairman) held no meeting during the year. This subcommittee does not contemplate further meetings until such time as a revision in the monograph on "Paper and Paperboard, Characteristics, Nomenclature, and Significance of Tests," is necessary.

Subcommittee IV on Fiberboard and Fiberboard Containers (R. L. Beach, chairman) has held one meeting during the year. Test procedures for the following are under consideration:

- Ring crush test,
- Adhesion test of component parts of corrugated board,
- Scuffing test,
- Immersion test,
- Delamination of plies,
- Bending quality of paperboard,
- Adhesive strength of seals and closures of packages,
- Insert resistance, and
- Water absorption of paperboard.

The Advisory Committee held two meetings during the year: on October 18, 1945, and March 1, 1946. During these meetings, the proposed reorganization of Committee D-6 was thoroughly discussed, and it was finally recommended that this matter be tabled for the present.

The Advisory Committee recommends the following additions to the Regulations of Committee D-6 for immediate adoption:

Article II.—Add the following as a new Section 2:

> SEC. 2. *Definition.*—Paper and paper products are those which do not lose their cellulose identity and do not contain a preponderance of other materials which would interfere with the methods of testing with apparatus commonly in use.

Article VII.—Add the following as a new Section 6:

> SEC. 6. *Official.*—Committee D-6 representatives on other standing committees of the Society and on outside organizations shall report the results of important actions affecting Committee D-6. Such a report shall be submitted in writing to the secretary of Committee D-6 for presentation to the Advisory Committee at its next annual meeting for appropriate action.

The Advisory Committee contemplates the formation of a new subcommittee for the development of specifications on paper. Bonds, ledgers, and index papers will be considered initially.

During the year, the officers of Committee D-10 on Shipping Containers and Committee D-6 met with A.S.T.M.

officials to consider the request of Committee D-10 to take over the work on corrugated containers now being handled by Subcommittee IV of Committee D-6. As a result of this meeting, it was agreed to turn over to Committee D-10 all of the methods, specifications, and work which Subcommittee IV has completed or is at present working on, which concern completed shipping containers. This included the Standard Method of Compression Testing of Corrugated Solid Fiber Boxes (D 642 – 43), Tentative Method of Drop Test for Shipping Containers (D 775 – 44 T), and Tentative Method of Drum Test for Containers in Small Revolving Hexagonal Drum Box-Testing Machines (D 782 – 44 T).

During the year, the officers of Committee D-9 on Electrical Insulating Materials and Committee D-6 met with A.S.T.M. officials to discuss the question of jurisdiction over vulcanized fiber. As a result of this meeting, it was agreed that the responsibility for standards on vulcanized fiber when used for structural purposes would be turned over to Committee D-6.

A Nominating Committee consisting of the chairmen of the subcommittees of Committee D-6, namely, R. L. Beach (chairman), W. R. Willets, and C. C. Heritage, submitted the following nominations for officers of the committee for the next two years, which will be voted on at the meeting of Committee D-6 held in June during the Annual Meeting of the Society:

Chairman, L. S. Reid.
Vice-Chairman, W. R. Willets.
Secretary, G. H. Harnden.

At the same time, the Nominating Committee recommended that the members-at-large of the Advisory Committee for the next two years be as follows:

K. P. Geohegan,
E. G. Ham,
G. M. Oleson, and
R. G. MacDonald.

This report has been submitted to letter ballot of the committee, which consists of 63 voting members; 55 members returned their ballots, of whom 54 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

L. S. REID,
Chairman.

G. H. HARNDEN,
Secretary.

APPENDIX

PROPOSED METHOD OF TEST FOR THE BACTERIOLOGICAL EXAMINATION OF PAPER AND PAPERBOARD[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method covers the procedure for the bacteriological examination of paper and paperboard. The procedure outlined represents in every case what may be considered at this time as minimum standard practice. Because of the exacting technique required in bacteriological procedures, they can be expected to yield reproducible results only in the hands of properly trained technicians. Likewise, more than usual care must be exercised in the collection and shipment of samples. If possible, this work should be done by a bacteriologist.

Apparatus

2. The apparatus shall consist of the following:

(*a*) *Balance.*—The balance shall be sensitive to 0.1 g. and should be equipped with a platform of a size to accommodate readily the Petri dishes used.

(*b*) *Colony Counter.*—Any one of several types of colony counters may be used. The Quebec, the Buck, and the Wolffhuegel colony counters have been found satisfactory. A hand tally for recording the count is recommended.

(*c*) *Containers for Dry Samples.*—Two single envelopes, one placed inside the other. The larger one may be 9 by 12 in. and the smaller 6.5 by 9.5 in. They should be made of heavy kraft paper, tested for ability to withstand breakdown due to embrittlement or charring during sterilization in the hot-air oven. After sterilization, the flap on the outer envelope shall be secured by Scotch tape. A folder of suitable size made from heavy kraft paper sterilized in the oven may also be used in place of the two single envelopes. After the paper sample has been placed inside the sterile folder, the latter is sealed with Scotch tape.

(*d*) *Cotton.*—Rolls of nonabsorbent cotton for making plugs.

(*e*) *Dilution Bottles.*—Narrow-mouthed, square-sectioned 8-oz. glass bottles fitted with screw caps for water blanks.

(*f*) *Disintegrators.*—These may be either the electrical type[2] or the ball-mill type. A kraft-paper cover shall be placed over each disintegrator cup or jar prior to its sterilization by steam heat.

(*g*) *Flaming Equipment.*—Either an alcohol lamp, a gasoline blowtorch or a Bunsen burner for flaming forceps, scissors, knives, mouths of sterile containers, etc.

(*h*) *Flasks.*—200-ml. Erlenmeyer flasks as containers for sterile media.

(*i*) *Forceps* for handling samples of paper. Each pair shall be placed in a separate kraft envelope made especially for this purpose, and then sealed with gummed tape preparatory to sterilization. Alternately, the forceps may be sterilized by flaming, in which case the envelope is unnecessary.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products. Published as information, June, 1946.

[2] Disintegrators such as the Stevens, Stevens Electrical Co., Racine, Wis., or the Berated, Berated Mfg. Co., Fostoria, Ohio, have been found satisfactory.

(*j*) *Incubator.*—A reliable incubator which will maintain a temperature of 37 ± 0.5 C. (98.0 ± 1.0 F.), to provide proper incubation conditions for the inoculated plates and tubes.

(*k*) *Knives.*—A paring knife of the type commonly used to cut fiberboard with its blade cut down to within 1.5 in. of the base. The knives shall be prepared for sterilization as described in Paragraph (*i*).

(*l*) *Petri Dishes.*—Petri dishes, 100 by 15 mm., preferably of Pyrex glass, are recommended for plating samples. They may be sterilized conveniently in copper boxes fitted with covers.

(*m*) *Pipettes.*—Two sizes of pipettes, 1.1-ml. milk-dilution type and 10-ml. ordinary type. Mohn 10-ml. pipettes whose tips have been cut off to give an opening about 3 mm. in diameter at the delivery end, are best for pipetting pulp suspensions. The pipettes shall be placed in metal boxes (preferably copper) to be sterilized.

(*n*) *Scissors.*—A satisfactory type scissors shall have a 4-in. cutting surface. Each pair shall be prepared for sterilization as described in Paragraph (*i*).

(*o*) *Sterilizers.*—Two types are needed, the particular style and size depending upon the needs of each laboratory as follows:

(*1*) A steam sterilizer, such as the pressure cooker or the autoclave, and

(*2*) A hot-air oven, preferably electric, although gas-heated ovens may be used.

In both cases, thermometers shall be used to determine the inside temperature during sterilization.

Chemicals and Media

3. (*a*) *Alcohol.*—Either methyl alcohol or 95 per cent denatured ethyl alcohol shall be used to sterilize equipment by flaming.

(*b*) *Culture Medium.*—A standard nutrient agar (Note) may be prepared from the essential ingredients according to the procedure outlined by the American Public Health Association[3]. After the medium is dissolved, the hot mixture shall be placed in Erlenmeyer flasks, in dilution bottles, or in test tubes, and plugged. It is then ready to be sterilized.

NOTE.—Difco dehydrated nutrient agar or equivalent is recommended.

Sterilization of Apparatus and Media

4. Depending upon the nature of the equipment to be sterilized, one of three methods shall be used as follows:

(*a*) *Steam Heat* (*Pressure Cookers and Autoclaves*).—The following shall be sterilized by heating for 20 min. at a minimum temperature of 121 C. (260 F.), corresponding to 15-psi. steam pressure: cups and jars for disintegrators, culture medium (unless other conditions are specified by the manufacturer of the medium), and water for dilutions.

(*b*) *Dry Heat* (*Electrically or Gas-Heated Ovens*).—The following shall be sterilized by heating for 4 to 5 hr. at a minimum temperature of 165 C. (330 F.): heavy kraft envelopes and folders, forceps, knives, Petri dishes, pipettes, and scissors. Care shall be taken to avoid overheating and consequent scorching of any paper containers or wrappers used on instruments that are being sterilized.

(*c*) *Flaming.*—Scissors, forceps, knives, and similar instruments shall be alternatively sterilized by flaming. Such apparatus shall be kept in denatured 95 per cent alcohol or in methyl alcohol while not in use. When needed for cutting or handling samples, the apparatus shall be removed from the liquid, the alcohol drained off for a few moments, then the excess alcohol burned off in the flaming equipment.

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[3] "Standard Methods for the Examination of Water and Sewage," Am. Public Health Assn., 1936, p. 201.

Sampling

5. Occasional single samples are inadequate, since the analyses reflect the conditions which prevailed only at the time when such samples were taken. The actual number of samples which should be taken depends largely on the reasons which prompt the analysis.

(*a*) *Sampling Lots of Paper or Paperboard.*—At least five representative rolls or skids shall be taken from 50,000-lb. lots. For smaller lots, at least three representative samples should be taken.

(*b*) *Sampling Paper or Paperboard on Machines.*—When a lot of paper or paperboard is being examined during its manufacture, at least 4 samples should be taken at 6-hr. intervals during each 24 hr. In some cases, it may be desirable to sample every 4 hrs.

(*c*) *Collection of Samples of Paper and Paperboard.*—Two sterile envelopes, one within another, shall be used for each sample. Every two weeks, a check shall be made on the technique of handling these sterile envelopes by sampling the inner envelopes as if they were paper samples, and testing for bacterial content.

Samples from rolls shall be taken as follows: Make a vertical cut with a sterile knife through several thicknesses on the surface of the roll near the center and parallel to the edge of the roll; make a second vertical cut parallel to the first and about 5 in. from the first cut; and make a horizontal cut across the bottom of the first two cuts, thus allowing the cut flap to spring out from the surface of the roll. Then carefully open the sterilized envelope, tear off and discard the top sheet of the flap, and slip the end of the cut flap of paper into the envelope. Then make a last horizontal cut across the top of the flap and about 8 in. from the bottom cut. The paper sample will then drop into the inner envelope, after which the outer envelope shall be sealed by means of Scotch tape. This will give a sample approximately 5 by 8 in. Two such samples shall be taken at different sections on the roll. This procedure may be adapted for sampling paper from skids, using flamed forceps for handling the samples of cut paper.

Procedure

6. Weighing, plating, and culturing of paper samples shall be done in a room which is relatively free of air currents and dust. The surface of the work table shall be sponged with a 5 per cent aqueous solution of phenol about 30 min. before plating.

(*a*) *Weighing Method.*—Each sample shall be weighed in a tared, sterile Petri dish placed on the balance as follows: Cut the envelope, in which the sample is received, along the flap by means of a sterile knife or sterile scissors. Open the envelope by pressing the sides, without touching the inner surface, and remove the paper sample with sterile forceps. Hold the sheet of paper in one hand, cut away with sterile scissors and discard the outer edges of the sheet. Make a series of cuts about $\frac{1}{4}$ to 1 in. apart, parallel to the side of the sheet and opposite the edge held in the hand. Remove the cover of the tared Petri dish and place it on another sterile dish, the cover of which is removed at the time of cutting the sample. Cut squares of paper directly into the dish by making a series of cuts perpendicular to those made previously. The particular type of disintegration equipment employed and the volume of liquid which it will handle satisfactorily will govern the amount of paper sample which is to be cut for disintegration. Therefore, cut sufficient paper to make a 1.0 per cent suspension of fibrous material after disintegration has been completed. Not less than 2.0 g. of paper sample should be used. Replace the cover on the Petri dish; check the weight and adjust the excess or deficiency to obtain the desired weight of sample. A tolerance of plus or minus 10 per cent is permitted in the weight of the sample.

(*b*) *Disintegration.*—Place the weighed sample and the necessary volume of sterile water in the disintegrator cup or jar. In adding material to the cup, do not touch the metal cap with the hands but lift it by grasping the paper hood which was placed over the cup before sterilizing. Lift both hood and metal cap only enough to permit access to the cup. If several paper samples are to be examined, cool the water blanks to prevent the temperature of the disintegrated sample from exceeding 45 C. (113 F.) during disintegration. Let the paper hood remain on the cup at all times to prevent possible contamination through the opening in the metal cap or around the top of the cup. Place the cup containing the weighed paper sample and sterile water in the mixing machine and run for approximately 6 min., or until disintegration of the paper is completed. Test the sterility of the cups periodically by adding sterile water, as when examining paper, and then culturing portions of the rinse water for controls. Make control tests on the technique of disintegration at frequent intervals by using a sample of sterilized paper and following the above procedure in detail. Plates containing such samples should be free from colonies of bacteria.

(*c*) *Plating and Incubating.*—In culturing a disintegrated paper sample, distribute with a sterile pipette a total of 10 ml. of the pulped mixture in approximately equal portions in five Petri dishes. Add to each plate 15 to 20 ml. of standard nutrient agar, cooled to about 45 C. (113 F.), then agitate the plates individually to disintegrate clumps of pulp and obtain an even distribution of the paper fibers in the medium. A reciprocating motion, similar to that on the wire screen of a Fourdrinier machine, is desirable. It is important that all lumps of pulp be broken up in order that the plates may be examined easily and accurately. Pour one control plate (without paper pulp) with each set of five plates, thus providing an agar control for each paper sample examined. After the plates have been agitated, place them on a level surface until the medium solidifies; then invert and incubate the plated samples at 37 ± 0.5 C. (98 ± 1 F.) for 48 hr. Do not stack the plates too closely in the incubator.

(*d*) *Counting Plate Cultures.*—After incubation for 48 hr. at 37 C., examine the cultures for the presence and number of colonies by holding the plates against a dark background, indirectly lighted. Plate counters are especially desirable. The amount of fibrous material distributed over a given area of plate surface has been found to be an important factor in the number of colonies observed in a given sample of paper. Therefore, it is important to follow carefully the directions regarding the pulp consistency (1.0 per cent), volume of sample plates (10 ml.), and the number and size of plates used. If the paper has been separated into individual fibers and mixed well in the medium, the colonies may be easily counted. If any particles are observed in the cultures which cannot be definitely identified as colonies, they should be examined microscopically. Plates with spreading colonies should be counted if at all possible; otherwise, they should be reported as "spreading colonies." Examine control plates also and make a record of the number of colonies found on the control of each paper sample.

Calculation of Results

7. Results for paper samples shall be expressed as the number of colonies per gram of air-dry paper examined. Since the consistency of the fibrous suspension made from such samples is 1.0 per cent,

there is present 1.0 g. of stock in each 100 g. of suspension. A total of 10 ml. of the pulped mixture (0.10 g. of actual paper) is cultured in five plates. Therefore, to obtain the number of colonies per gram of paper, multiply the total number of colonies found by 10.

Report

8. Report the average number of colonies per gram according to the following method[4]:

Number of Colonies per Gram	Report to Nearest
1 to 50	1
51 to 100	5
101 to 250	10
251 to 500	25
501 to 1 000	50
1 000 to 10 000	100
10 001 to 50 000	500
50 001 to 100 000	1 000
100 001 to 500 000	10 000
500 001 to 1 000 000	50 000
1 000 001 to 10 000 000	100 000

As explained in Section 7, the count of any particular sample of paper is obtained by multiplying the total count on the plates by 10. However, when the counts of several samples are to be averaged, the logarithmic method of averaging is recommended (Note). This method of averaging tends to minimize the effect of occasional high counts which may have resulted from some transitory condition. Details concerning logarithmic averages have been published.[5]

NOTE: *Example.*—An example of this type of averaging follows:

Count	Logarithm
1 00	2.0
1 000	3.0
10 000	4.0

The logarithmic average of these three counts would be 3.0, representing 1000 colonies per gram.

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[4] "Standard Methods for the Examination of Water and Sewage," Am. Public Health Assn., 1936, p. 209.

[5] "Mills Ordinance and Code," *Bulletin 220*, U. S. Public Health Service (1939).

REPORT OF COMMITTEE D-7*

ON

WOOD

Committee D-7 on Wood held one meeting during the year in Chicago, Ill., on March 12, 1946, at which the status of work under way and plans for the coming year were discussed.

At this meeting of the committee it was voted to establish a new subcommittee on wood poles, to be designated "Subcommittee VII on Wood Poles." This action was taken because of the increasing demand for wood poles involving a consideration of and specifications for additional species other than those which have been most commonly used. This action will still further broaden the field of work of the committee and enable it better to meet the range of problems in its field.

At the meeting of the committee the present officers were unanimously reelected for the ensuing term of two years.

A summary of the work under way and further plans of the several subcommittees is discussed under the headings of the various subcommittees.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Specifications for Timber (L. J. Markwardt, chairman) is planning some revision in the Standard Specifications for Structural Wood Joist and Plank, Beams and Stringers, and Posts and Timbers (D 245 – 37) to provide material suitable for such purposes as tension members in trusses and to convert the present grade for beams and stringers, and joists and planks into cutting grades. Some suggestions for revision of the Standard Specifications for Round Timber Piles (D 25 – 37) have been received and will be considered.

Subcommittee IV on Wood Paving Blocks (W. H. O'Brien, chairman) has developed a preliminary draft of tentative specifications for wood block flooring for interior use. The preliminary draft is undergoing further revision to meet requirements found to be most satisfactory on the part of users and manufacturers. It is planned to present the specifications for publication as tentative during the coming year.

Subcommittee V on Methods of Preservative Treatment of Timber (A. L. Kuehn, chairman) is considering the advisability of revising and bringing up to date specifications on methods of preservative treatment so that they will conform with latest practices.

Subcommittee VI on Timber Preservatives (W. P. Arnold, chairman) will give major consideration to the preparation of specifications for other extensively used preservatives than those now covered by A.S.T.M. Standards, and has scheduled for review and revision a number of the present A.S.T.M. methods and specifications as follows:

Standard Methods of Test for:[1]

Chemical Analysis of Zinc Chloride (D 199 – 27),
Insoluble Matter in Creosote (D 367 – 33),
Specific Gravity of Creosote (D 368 – 33),
Specific Gravity 38/15.5 C. of Creosote Fractions (D 369 – 33),
Water in Creosote (D 370 – 33), and

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] 1944 Book of A.S.T.M. Standards, Part II.

Standard Specifications for:

Zinc Chloride (D 432 – 39).

Subcommittee IX on Methods of Testing (L. J. Markwardt, chairman) has made marked progress in the revision of the Standard Methods of Testing Small Clear Specimens of Timber (D 143 – 27). This revision will be completed during the year. Work is also progressing on the development of a secondary standard for methods of testing wood based on specimens 1 by 1 in. in cross-section as against the 2 by 2-in. size called for in the present standard. The need of the secondary standard is becoming increasingly important to permit the more efficient evaluation of the properties of second growth material which involves smaller trees than virgin growth and for studying the properties of other species which do not attain sufficient commercial size to yield adequate sampling as called for in the present Standard Method D 143.

Subcommittee X on Nomenclature and Definitions.—With the resignation of C. J. Hogue, the chairmanship of this subcommittee remains to be filled. During the coming year completion of the revision of tree names in the Standard Definitions of Terms Relating to Timber (D 9 – 30) is expected.

Subcommittee XI on Moisture Content of Timber (M. E. Dunlap, chairman) contemplates carrying on research specifically directed to the use of moisture meters which will lead to the preparation of specifications for their use. Included in the problems under study, are such factors as the evaluation of the effect of glue lines on moisture meter readings on laminated timber.

Mr. Dunlap reported the development of a satisfactory method at the Forest Products Laboratory for quickly determining the moisture content of wood treated with fire-retardant chemicals. Considerable difficulty has been experienced in connection with the determination of the moisture content of treated wood. For this type of material the usual oven-dry method is not accurate and moisture meters do not give reliable results. The new method developed by Mr. Dunlap is based on the dew-point principle and gives promise of solving the problem of determining the moisture content of chemically treated wood. The method involves placing small sections of the sample in a glass jar of special design and after moisture equilibrium of the air has been reached, the moisture content can be determined with surprising accuracy. About 20

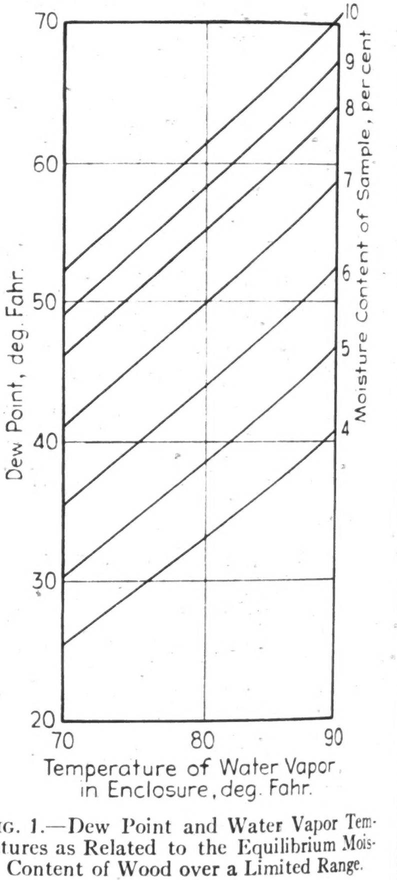

FIG. 1.—Dew Point and Water Vapor Temperatures as Related to the Equilibrium Moisture Content of Wood over a Limited Range.

min. is required for a determination. A special chart shown in Fig. 1 has been prepared for converting the reading to the usual percentage moisture content, based on the dry weight of the wood. This development elicited great interest among the committee members interested in treated wood.

The apparatus consists of a clear glass jar with a tight cover to contain the sample, a dew-point instrument, a thermometer to register the temperature of the water vapor, and vessels to supply chilled water for cooling the dew-point instrument.

The dew-point measurement is a well-known physical determination and is found by cooling a highly polished metal surface to the temperature at which a cloud of water vapor condenses on and leaves the surface.

The chart (Fig. 1) gives directly the moisture content of the sample when the temperature of the water vapor and dew point are known, for the limited range of moisture content encountered in tests of treated wood. The chart is based on the well-established relation between moisture content, relative humidity, and temperature.

Subcommittee XII on Fire-Retardant Wood (W. H. Fulweiler, chairman) has been active during the year in studying problems relating to methods of testing fire-retardant wood, the significance of results, and the correlation of the different methods. One of the basic problems is that of sampling; and complicating the sampling for fire test is the fact that some pieces of wood treat readily on all sides, while others do not treat uniformly. The problem of sampling involves, furthermore, such questions as whether the test is to evaluate lumber that is completely treated and involves relatively small sizes, or to evaluate large timbers that are not completely treated through the piece. While much has been accomplished, the work has not progressed far enough to lead to the completion of specific conclusions and recommendations.

STANDARDS SIX YEARS WITHOUT REVISION

The following standards under the jurisdiction of Committee D-7 have now been published for six years or longer without revision. These standards have been carefully reviewed and they should all be continued without any changes. Most of them are now the accepted standards of other engineering groups and Governmental bureaus and will probably not require any changes for some time to come:

Standard Specifications for:

Wooden Paving Blocks for Exposed Pavements (D 52 - 20),
Creosote (D 390 - 36),
Creosote - Coal Tar Solution (D 391 - 36),

Standard Methods of:

Sampling and Testing Creosote (D 38 - 33),
Test for Coke Residue of Creosote (D 168 - 30),
Static Tests of Timbers in Structural Sizes (D 198 - 27),
Test for Distillation of Creosote (D 246 - 42),
Standard Definition of Terms Relating to Timber Preservatives (D 324 - 41), and
Standard Volume and Specific Gravity Correction Tables for Creosote and Coal Tar (D 347 - 33).

The following standards under the jurisdiction of Committee D-7 are now either undergoing revision or are included in plans for revision during the coming year by the respective subcommittees:

Standard Specifications for:

Round Timber Piles (D 25 - 37),
Structural Wood Joist and Plank, Beams and Stringers, and Posts and Timbers (D 245 - 37),
Zinc Chloride (D 432 - 39),

Standard Methods of:

Testing Small Clear Specimens of Timber (D 143 - 27),

Chemical Analysis of Zinc Chloride (D 199 – 27),
Test for Insoluble Matter in Creosote (D 367 – 33),
Test for Specific Gravity of Creosote (D 368 – 33),
Test for Specific Gravity 38/15.5 C. of Creosote Fractions (D 369 – 33),
Test for Water in Creosote (D 370 – 33), and
Definitions of Terms Relating to Timber (D 9 – 30).

This report has been submitted to letter ballot of the committee, which consists of 49 members; 46 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

HERMANN VON SCHRENK,
Chairman.

L. J. MARKWARDT,
Secretary.

REPORT OF COMMITTEE D-8*

ON

BITUMINOUS WATERPROOFING AND ROOFING MATERIALS

Because of war conditions, Committee D-8 on Bituminous Waterproofing and Roofing Materials held no meetings and made no report to the Society in 1945. However, during that year Committee D-8 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for Asphalt Roofing Surfaced with Coarse Mineral Granules (D 249 - 44 T), and

Revision of Emergency Alternate Provisions for Tentative Specifications for Asphalt Roofing Surfaced with Coarse Mineral Granules (EA - D 249b).

These recommendations were accepted[1] by the Administrative Committee on Standards on August 27, 1945, and appear in the 1945 supplement to Book of A.S.T.M. Standards, Part II.

During 1945, a new Subcommittee XIII on Tests for Stain Properties of Bituminous Materials has been created under the chairmanship of A. B. Hersberger, which is now engaged in:

(*1*) A cooperative study and investigations required in order to standardize a stain test based on the Schweyer-Howell Test.

(*2*) The selection and study of asphalts and test methods connected with the problem of staining of paint coatings by asphalts used in the construction of floor coverings.

Subcommittee XII on Tests for Mineral Surfacing Materials, under the chairmanship of H. R. Gundlach, has prepared specifications for grading of roofing granules covering three grades now being used in the asphalt roofing industry, which, it is hoped, may soon be presented to the Society through the Administrative Committee on Standards for publication as tentative.

Prior to a meeting of Committee D-8 at Pittsburgh, Pa., on February 27, 1946, its subcommittees reviewed all of the standards and tentatives for which they are responsible, and at that meeting action was taken by Committee D-8 on the recommendations in this report.

I. REVISION OF TENTATIVES

In order to bring two tentative specifications into general conformity with other roofing specifications, Committee D-8 recommends that they be revised as follows:

Tentative Specifications for Asphalt Roofing Surfaced with Powdered Talc or Mica (**D 224 - 44T**):[2]

Table I.—Place the ninth requirement as the sixth requirement in this table and change to read as follows by the omission of the words in brackets: "Weight of any 108 sq. ft. [roll] of [complete] roofing in the shipment."

Tentative Specifications for Asphalt

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

[1] In submitting these recommendations to the Administrative Committee on Standards, Committee D-8 reported the following results of the letter ballot vote of a total of 39 ballots returned from a committee membership of 62: D 249, affirmative 31, negative 0, ballots marked "not voting" 8; EA - D 249b, affirmative 32, negative 0, ballots marked "not voting" 7.

[2] **1944 Book of A.S.T.M. Standards, Part II.**

Roofing Surfaced with Coarse Mineral Granules (**D 249 – 45 T**):[3]

Title.—Delete the word "Coarse."

Section 6 (*c*).—Change from its present form: namely,

> The reverse side of the sheet shall have the asphalt coating and surfacing applied uniformly to the edges.

to read as follows:

> The asphalt coating and the surfacing shall be applied uniformly over the entire reverse side of the sheet.

Table I.—Change the requirements for area to read as follows by the addition of the italicized figures and the omission of those in brackets: "Area. . .The average area of the rolls examined shall contain sufficient roofing to cover 100 sq. ft. of roof surface (108 sq. ft. with 2 in. or no bare lapping edge or [111] *114* sq. ft. with [3] *4* in. lapping edge)."

Change the minimum requirements for net average weight per roll to read as follows by the addition of the italicized figures and the omission of the figure in brackets:

	Weight per Roll, net average, min., lb.
No bare edge	83.0
2 [or 3]-in. bare edge	81.0
4-in. bare edge	*83.0*

Place the last requirement as the sixth requirement, and change to read as follows by the omission of the words in brackets: "Weight of any 108 sq. ft. [roll] of roofing in the shipment ([coarse] granule-surfaced portion)."

Section 10 (*a*).—Change the first sentence to read as follows by the addition of the italicized figures and the omission of those in brackets: "The roofing shall be put up in rolls of either 108 sq. ft. when made with a 2-in. or no bare-lapping edge, or [111] *114* sq. ft. when made with a [3] *4*-in. bare lapping edge."

Section 12 (*a*).—Change the second sentence of item (*3*) of this section to read as follows by the addition of the italicized figure and the omission of the figure in brackets: "Where the roofing is intended for application by the concealed nailing method and is provided with a [3] *4*-in. selvage, 150 nails per square shall be furnished on a basis of 36-in. roofing."

Section 12 (*b*).—Change the fifth, sixth, and seventh sentences of item (*2*) of this section to read as follows by the addition of the italicized figures and the omission of those in brackets: "The test specimen shall consist of two strips each 6 in. in length and 1 in. in width cut from the roofing, and one of the strips shall include a [3] *4*-in. bare lapping edge. The specimen shall be prepared for testing by covering the bare lapping edge of the one strip with [1.5] *2* g. of cement, spreading it evenly, and then allowing the strip to remain undisturbed for 10 min. at a temperature of 70 to 90 F. (21 to 32 C.). The reverse side of the other strip shall then be placed upon the cement-coated lapping edge in such a manner as to form a complete specimen [9] *8* in. in length by 1. in. in width that includes a regular [3] *4*-in. cemented lap."

Tentative Methods of Sampling Bituminous Materials (**D 140 – 41 T**).[2]—These methods are under the joint jurisdiction of Committee D-8 and Committee D-4 on Road and Paving Materials. Committee D-8 joins with Committee D-4 in recommending the following revision of these methods:

Section 8 (*a*).—Change the third and fourth sentences to read as follows by the addition of the italicized words and the omission of the words and figures in brackets:

> This pipe shall be provided with a plug cock and shall discharge into a [receiving drum of 50-gal. capacity] *sample receiver*. The plug cock

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[3] 1945 Supplement to Book of A.S.T.M. Standards, Part II.

shall be so adjusted that there is a steady continuous flow of bituminous material through it and [shall be] so regulated that the *sample* receiver will fill *to the required amount* in the same time that is required to make the entire pumping.

Section 13.—Change this section to read as follows by the addition of the italized words and figures:

13. Solid bituminous materials in crushed fragments or powder, such as gilsonite, grahamite, etc., shall be sampled in accordance with the Standard Method of Sampling Coal for Analysis (A.S.T.M. Designation: D 21), *except that the gross sample may be less than 1000 lb. but not less than 50 lb. when knowledge of the uniformity of the solid bituminous material in the shipment warrants.*

II. ADOPTION OF TENTATIVE AS STANDARD

Tentative Specifications for Asphalt Shingles Surfaced with Coarse Mineral Granules (**D 225 - 44 T**).[2]—Committee D-8 recommends that these tentative specifications, with the following editorial revisions, be approved for reference to letter ballot of the Society for adoption as standard:

Title.—Delete the word "coarse."

Section 3.—Add the following as the first sentence of this section: "The felt shall be a roofing felt produced by "felting" vegetable or animal fibers or mixtures thereof."

Table I.—Change the fourth item to read as follows by the addition of the italicized words: "Weight (net average *of package inspected*) of finished shingles per 108 sq. ft."

III. REVISION OF STANDARDS, IMMEDIATE ADOPTION

As the proposed revisions of the following standards are mainly editorial in nature, and are intended to bring them into conformity with other approved forms, it is recommended that they be incorporated immediately in these standards. The committee accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society.

Standard Specifications for Wide Selvage Asphalt Roofing Surfaced with Coarse Mineral Granules (**D 371 - 44**):[2]

Title.—Delete the word "Coarse."

Section 6 (c).—Change the last sentence to read as follows by the addition of the italicized words and the omission of those in brackets: "If coated it may be [surfaced] *covered* with a [small amount of mineral matter] *suitable material* to prevent *the roofing from* sticking in the [roll] *package.*"

Table I.—Change the last sentence of the fourth requirement to read as follows by the addition of the italicized figure and the omission of the one in brackets: "The granular surfacing shall not slide more than [$\frac{1}{4}$] *1/16* in. when suspended vertically."

Place the last requirement as the sixth requirement in this table and change to read as follows by the omission of the word in brackets: "Weight of any 108 sq. ft. [roll] of roofing in shipment . . . ."

Standard Specifications for Asphalt Siding Surfaced with Coarse Mineral Granules (**D 699 - 44**):[2]

Title.—Delete the word "Coarse."

Section 3.—Add the following as the first sentence of this section: "The felt shall be a roofing felt produced by "felting" vegetable or animal fibers or mixtures thereof."

Table I.—Place the last requirement as the fifth item in this table.

IV. STANDARDS AND TENTATIVES CONTINUED WITHOUT REVISION

Standard Specifications for Asphalt for Use in Constructing Built-Up Roof Coverings (**D 312 - 44**).[2]—No immediate revision of this standard is contemplated, although certain suggestions for changes in softening point and ductility requirements are under consideration.

Tentative Methods of Testing Asphalt Roll Roofing, Cap Sheets and Shingles (**D 228 - 44 T**).[2]—Work is now being conducted on probable improvements in some of the test methods.

Tentative Specifications for Asphalt-Saturated Asbestos Felts for Use in Waterproofing and in Constructing Built-Up Roofs (**D 250 - 44 T**),[2] and for *Asphalt-Saturated and Coated Asbestos Felts for Use in Constructing Built-Up Roofs* (**D 655 - 44 T**).[2]—There are several differences between these two specifications and those under consideration by the Federal Specifications Board, which should be studied before the committee is prepared to recommend them for adoption as standard.

Tentative Specifications for Coal-Tar Pitch for Steep Built-Up Roofs (**D 654 - 42 T**).[2]—Studies of the softening point range and requirements for resistance to slide are being conducted.

Tentative Specifications for Asphalt for Dampproofing and Waterproofing (**D 449 - 42 T**).[2]—It is considered advisable to maintain these specifications as tentative, pending an attempt to harmonize them with proposed requirements of the American Railway Engineering Association.

Tentative Recommended Practice for Accelerated Weathering Test of Bituminous Materials (**D 529 - 39 T**).[2]—A revision of this test will probably be recommended in the near future.

The recommendations appearing in this report have been submitted to letter ballot of the committee which consists of 60 voting members, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. REVISION OF TENTATIVES			
Spec. for Asphalt Roofing Surfaced with Powdered Talc or Mica (D 224 - 44 T)	33	0	8
Spec. for Asphalt Roofing Surfaced with Coarse Mineral Granules (D 249 - 45 T)	34	0	7
Methods of Sampling Bituminous Materials (D 140 - 41 T)	33	0	8
II. ADOPTION OF TENTATIVE AS STANDARD			
Spec. for Asphalt Shingles Surfaced with Coarse Mineral Granules (D 225 - 44 T), as revised	33	1	7
III. REVISION OF STANDARDS, IMMEDIATE ADOPTION			
Spec. for Wide Selvage Asphalt Roofing Surfaced with Coarse Mineral Granules (D 371 - 44)	34	0	7
Spec. for Asphalt Siding Surfaced with Coarse Mineral Granules (D 699 - 44)	32	0	9

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, J. S. Miller.
Vice-Chairman, E. H. Berger.
Secretary, H. Clay Howell.

This report has been submitted to letter ballot of the committee, which consists of 60 voting members; 41 members returned their ballots, of whom 38 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. S. MILLER,
Chairman.

PRÉVOST HUBBARD,
Secretary.

REPORT OF COMMITTEE D-9[*]

ON

ELECTRICAL INSULATING MATERIALS

Committee D-9 on Electrical Insulating Materials held two meetings during the year and will hold a third during the Annual Meeting of the Society in June. These meetings were held in Atlantic City, N. J., on October 11 and 12, 1945, and on March 7 and 8, 1946.

At these meetings actions were taken which resulted in several new methods, revisions in existing standards, and other changes in some of the standards for which Committee D-9 is responsible. Some projects which are still under way are mentioned later in this report.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1945 Annual Meeting, Committee D-9 presented to the Society through the Administrative Committee on Standards the recommendation that the Tentative Specifications for Molds for Test Specimens of Molding Materials Used for Electrical Insulation (D 647 – 42 T) be revised. This recommendation was accepted[1] by the Standards Committee on December 8, 1945, and the revised tentative appears in the 1945 Supplement to Book of A.S.T.M. Standards under the designation D 647 – 45 T.

On December 10, 1945, the Standards Committee accepted[1] the joint recommendation of Committee D-20 on Plastics and Committee D-9 that the Tentative Specifications for Phenolic Molding Compounds (D 700 – 43 T) be revised.

This revised tentative appears in the 1945 Supplement to Book of A.S.T.M. Standards under the designation D 700 – 45 T.

RECOMMENDATIONS AFFECTING STANDARDS

As the result of the year's work, the committee is submitting four new tentatives, revisions in seven tentatives, revision of standard and reversion to tentative, adoption of a tentative as standard, revisions of three standards for immediate adoption, and the adoption as standard of four tentative revisions of standards. One emergency standard was recommended for publication as tentative at the time of the Annual Meeting.

These recommendations have been submitted to letter ballot of the committee, which consists of 118 voting members, with the results shown in Table I.

ACTIVITIES OF SUBCOMMITTEES

Some of the projects now in progress are briefly mentioned in the following summary of committee activities:

Subcommittee I on Insulating Varnishes, Paints, and Lacquers (H. J. L. Cotton, chairman).—The subcommittee submitted a revision of Standard Methods of Testing Varnishes Used in Electrical Insulation (D 115 – 41), which is

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[*] Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] In submitting these recommendations to the Standards Committee, Committee D-9 reported the following results of the letter ballot vote of a total of 79 ballots returned from a membership of 118: **D 647**, affirmative 34 negative 0, ballots marked "not voting" 45; **D 700**, affirmative 27, negative 1, ballots marked "not voting" 51.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. New Tentatives			
Methods of Testing Nonrigid Polyvinyl Tubing	17	0	41
Method of Test for Dielectric Strength of Insulating Oil of Petroleum Origin	38	0	40
Method of Test for Inorganic Chlorides and Sulfates in Insulating Oils	33	0	45
Spec. for Communication and Signal Pin-Type Lime-Glass Insulators	23	0	62
II. Revisions of Tentatives			
Methods of Testing Molded Materials Used for Electrical Insulation (D 48 - 45 T)	39	0	39
Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (D 150 - 45 T)	49	1	28
Methods of Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 - 45 T)	19	0	39
Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (D 295 - 44 T)	24	1	33
Test for Power Factor and Dielectric Constant of Natural Mica (D 351 - 42 T)	36	0	49
Method for Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 - 45 T)	47	1	30
Specifications for Laminated Thermosetting Materials (D 709 - 44 T)	32	0	46
Specifications for Low and Medium Voltage Pin-Type Lime-Glass Insulators (D 730 - 43 T)	13	1	47
III. Revisions of Standards, Immediate Adoption			
Methods of Test for Insulation Resistance of Electrical Insulating Materials (D 257 - 45)	53	0	25
Methods of Testing Pin-Type Lime-Glass Insulators (D 468 - 42)	12	1	48
Test for Acetone Extraction of Phenolic Molded or Laminated Products (D 494 - 41)	34	0	51
IV. Revision of Standard and Reversion to Tentative			
Methods of Testing Varnishes Used in Electrical Insulation (D 115 - 41)	22	0	36
V. Adoption of Tentative as Standard			
Methods of Test for Power Factor and Dielectric Constant of Natural Mica (D 351 - 42 T)	16	0	42
VI. Adoption as Standard of Tentative Revisions of Standards			
Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 - 43)	31	0	30
Methods of Testing Tubes Used in Electrical Insulation (D 348 - 42)	24	1	36
Methods of Testing Laminated Round Rods Used in Electrical Insulation (D 349 - 42)	24	1	36
Methods of Test for Grading and Classification of Natural Mica (D 351 - 38)	12	0	46

appended hereto.[2] Section B which handled the revision will continue its work since there are additional editorial changes that were suggested by members at the time of voting. As a result of the revised standard and subsequent discussion, there is being formed a new Section G on Heat Endurance Tests. The former portion of the methods known as "heat endurance test" has been renamed "resistance to embrittlement by heat."

Work is being continued in Section C on the problem of tests for deep-drying varnishes. A number of laboratories are now cooperating in the round-robin test under definite laboratory procedure.

There has been established by Section D on Resistance to Acid and Alkali and Sea Water that the smoothness of the brass rod used in the test required by the Navy Department specifications was not a factor of influence in the test procedure. A rod roughened by steel wool gave as good results as a rod polished under the most careful conditions.

Section F is continuing the work of testing various cup designs in the determination of set time of thermosetting varnishes.

Subcommittee III on Plates, Tubes, Rods, and Molded Materials (G. H. Mains, chairman).—With the retirement of W. A. Evans from active work with the committee after many years of guidance in Subcommittee II and several years as chairman, the Advisory Committee has combined the work of the two subcommittees under a joint committee known as Subcommittee III on Sheets, Rods, Tubes, and Molded Materials. The members will greatly miss the wise counsel and energetic leadership of Mr. Evans.

During the past year the subcommittee prepared the new Tentative Method of Test for Nonrigid Polyvinyl Tubing[3]

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[2] This revised method was accepted by the Society and appears in the 1946 Book of A.S.T.M. Standards, Part III-B.

[3] These methods were accepted as tentative by the Society and appear in the 1946 Book of A.S.T.M. Standards, Part III-B.

A revision of the Tentative Methods of Testing Molded Materials Used for Electrical Insulation (D 48 – 45 T) has been completed which covers the addition of several paragraphs on the utility of the tests described in these methods.

The tentative revisions of Standard Methods D 229 – 43 with reference to the tests for tensile, compressive, and dielectric strengths have been recommended for adoption as standard; also the tentative revisions of Standards D 348 – 42 and D 349 – 42 which concern dielectric strength parallel to laminations.

An editorial change was made last year in Methods D 229 involving a modification of Section 3 to bring the requirements for conditioning into accord with Methods D 618 as revised last year. This revision was approved by letter ballot of Committee D-9 which consists of 118 members; 61 members returned their ballots, of whom 30 voted affirmatively, 0 negatively, and 30 members marked their ballots "not voting."

Some data have been presented on the effect of specimen size and conditioning procedures on measurment of insulation resistance and on the effect of high and low temperatures on power factor of sheet insulating materials. This work is being continued. A revision of the Rockwell hardness method to make it applicable to the complete range of plastics is being studied in cooperation with Subcommittee II of Committee D-20 on Plastics. A further study of the method of tension test for rods and tubes is being conducted in cooperation with Subcommittee XIII and Committee D-20; also, a new method of testing bonding strength of laminated materials and vulcanized fibre. Active work is under way on the preparation of specifications for nonrigid polyvinyl tubing, making use of the test methods just adopted. Electrical properties of the various thermoplastic molded materials and sheets are being investigated with a view of establishing specifications for certain electrical grades of these materials in cooperation with Committee D-20.

In accordance with the recommendation of the special joint Committee on Jurisdiction of Vulcanized Fibre, composed of representatives of Committee D-6 on Paper and Paper Products and Committee D-9, close cooperation will be maintained between this subcommittee and Committee D-6 in order to avoid any duplication of work.

Subcommittee IV on Liquid Insulation (E. A. Snyder, chairman).—Correlation tests to determine the relation of the sludge test values determined as described in tentative Method D 670 – 42 T with actual service life of insulating oil in transformers are being continued by approximately 20 cooperating laboratories. Nine of a bank of twelve transformers are being operated by a cooperating utility company under carefully controlled and supervised conditions. Three of these twelve transformers have not yet been put into operation. Two other utility companies are operating three transformers each under controlled conditions. A complete record of the operation of the transformers is being kept and samples of oil being drawn from the units every six months for examination and sludge tests. It is hoped that as a result of this work the subcommittee will be able to determine whether Tentative Methods D 670, for determining the sludging characteristics of oil, will actually differentiate between good and bad oils and correlate with actual service life obtained with these oils in transformers.

To date, the oils have not been in service long enough so that their service life quality can be established. The transformers have been in operation for only about one year. There are indications in the transformers operated by one utility company that the first-, second-, and third-grade oils as originally graded by the sludge test are showing pronounced differences in the rate of deterioration. Test data on these oils from the cooperating laboratories, however, show a wide variation not only on sludge tests but even on the simplest tests such as color and acid.

This work will, of course, continue and as indicated in the 1945 report,[4] it is anticipated that it will require approximately five years of operation in order to obtain sufficient data on which to base conclusions.

The reorganization of methods of test for insulating oils, referred to in the 1945 report, is proceeding as rapidly as possible and satisfactory progress is being made. A proposed tentative revision of Standard Methods D 117 is ready to be presented for letter ballot of Subcommittee IV. These methods refer to the individual test methods applicable to insulating oils and cite the A.S.T.M. serial designation number applying to each individual method. It then gives statements on definition of the method and significance of the tests.

Separate A.S.T.M. methods on the dielectric strength test and method for determining presence of inorganic salts (chlorides and sulfates) have been submitted to Committee D-9 for approval as new tentative methods.[3] A complete new individual method on sampling of insulating oils has been prepared and submitted to Subcommittee IV for letter ballot approval as a new tentative method. Individual test methods for power factor of insulating oils and for sulfur corrosion test are in the process of preparation and will soon be ready for Subcommittee IV letter ballot.

The section that is working on the development of a method of test to determine minute traces of water or moisture in insulating oils has been conducting a series of round-robin tests during the past year. Most of the work was devoted to studying the Karl Fischer method and the Doble method. Further work will have to be done before Subcommittee IV is ready to recommend a proposed tentative method of test.

The Doble method is described in a paper "Water Determination in New and Used Insulating Oils by Doble Method,"[5] being presented at this Annual Meeting of the Society. The Karl Fischer method has been adequately described in the literature and the following comments concerning these references will be of interest. The nature of the Karl Fischer reagent, including preparation, standardization, and chemical reaction taking place, is well explained by Fischer[6] and by Smith, Bryant, and Mitchell.[7] In light-colored solutions little difficulty is encountered in the colorimetric use of this reagent. However, in order to include highly colored solutions Almy, Griffin, and Wilcox,[8] and Aepli and McCarter,[9] have developed and adequately described the electrometric method using the Karl Fischer reagent. It is important to note, as explained by the above authors, that precautions must be taken to exclude atmospheric moisture during sampling and moisture determinations.

During the year it was determined that the present method for determining corrosive sulfur in insulating oils is not severe enough to eliminate marginal oils which contain traces of free sulfur that could be harmful to electrical apparatus. A new method which seems to be severe enough to eliminate all questionable oils has been developed and will be submitted to Subcommittee IV for letter ballot as a new proposed tentative method of test.

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[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 275 (1945).

[5] See p. 1475.

[6] *Angewandte Chemie*, Vol. 48, pp. 394–396 (1935).

[7] *Journal* Am. Chemical Soc., Vol. 61, p. 2407 (1939).

[8] *Industrial and Engineering Chemistry*, Anal. Ed., Vol. 12, p. 392 (1940).

[9] *Industrial and Engineering Chemistry*, Anal. Ed., Vol. 17, p. 316 (1945).

A new section has been organized to consider the matter of preparing specifications for insulating oils. This section has decided to develop a recommended practice to govern the purchase of insulating oils. The development of a recommended practice procedure involves the following steps:

1. The preparation of a screening specification to be used for selecting a limited number of oils which might be suitable for the required service.
2. The establishment of a recommended practice procedure to be followed by the purchaser of insulating oils which would embrace the following:
   (*a*) Procurement of proposed oils meeting the screening specification mentioned in item *1*, with copy of vendor's guaranteed characteristics of these oils.
   (*b*) Purchaser's laboratory examination of these samples to determine which might meet his requirements.
   (*c*) Purchaser's performance tests on trial orders of those samples selected according to item (*b*), to find out which of those will actually prove satisfactory in service.
   (*d*) On the basis of item (*c*) purchaser to prepare a list of approved suppliers with appropriate identification of the approved products by brand name, code number, or otherwise.

During the past few years methods of test to determine interfacial tension of insulating oils have been receiving considerable attention by industry. In view of the growing interest in this method of test, Subcommittee IV has established a new section to study the method and to develop a satisfactory procedure for its operation. This section will also endeavor to study the value of the test when used in determining the quality of insulating oils.

Subcommittee V on Ceramic Products (K. G. Coutlee, chairman).—Revisions in the Standard Methods of Testing Pin-Type, Lime-Glass Insulators (D 468 – 42), Tentative Specifications for Low and Medium Voltage Pin-Type, Lime-Glass Insulators (D 730 – 43 T) are recommended. The publication of Emergency Specifications for Communication and Signal Pin-Type, Lime-Glass Insulators (ES-41) with minor revisions, as a tentative specification is recommended.

The study of the factors involved in the determination of loss factor of steatite after immersion in water has reached a point where preliminary recommendation can be made which should reduce the variations now encountered when using the test procedure specified in Joint Army and Navy Specification JAN I-10. These recommendations have been forwarded to the Signal Corps for their consideration.

The sections of Subcommittee V have been reorganized and recently developed ceramic products have been added to the wide variety of ceramic products under the jurisdiction of Subcommittee V. Committee D-9 is being canvassed for members for the newly organized sections.

Work on the development of test methods for glass bonded mica is being started.

Subcommittee VI on Solid Filling and Treating Compounds (R. H. Titley, chairman).—The Section on Oil Solubility of Solid Filling and Treating Compounds has prepared and circulated to the members of Subcommittee VI a report on the work done.

A questionnaire, for the purpose of obtaining information relative to the writing of specifications for solid filling compounds and the revision of Standard Methods D 176, has been circulated to the members of Subcommittee VI and other interested consumers and manufacturers during the past year.

Statements on significance of tests have been prepared and approved by Subcommittee VI and copies have been forwarded to Subcommittee XI for approval.

Subcommittee VII on Insulating Fabrics (R. W. Chadbourn, chairman).—Progress has been made during the past year in the investigation of the slow-rate-of-rise dielectric strength test intended to replace the present step-by-step test for varnished cloth. Considerable test data have been accumulated, and the proposed method has met with favor in a number of laboratories. Simple modification of the present motor-driven voltage control has been evolved to include this test.

Data are being accumulated slowly on dielectric breakdowns on tapes subjected to 12 per cent elongation.

Statements on significance of tests are under consideration for treated sleeving. The statements developed for varnished cloth have been submitted to Subcommittee XI for comments.

The Section on Glass Fabrics has decided to divorce the test methods for glass fabrics from Tentative Methods D 295 and incorporate them in separate Tentative Methods of Testing Varnished Glass Fabrics and Varnished Glass Fabric Tapes. This move was necessitated by the difference in treatment of the two classes of material, due chiefly to different temperature limitations. Revised test methods have been written for conditioning, thickness, breaking strength, dielectric strength, power factor and dielectric constant, insulation resistance, and resistance to oil. Other tests will be studied and added when approved.

The newly-formed section on Electrical Adhesive Tapes has been very active during the year. Extensive investigations are being made of a number of pertinent properties, including thickness, adhesivity, effect of heat and moisture, tear resistance, tensile strength, fungus resistance, metal corrosion, electrolytic corrosion, and effect of varnishes, solvents, and oils.

Subcommittee VIII on Insulating Paper (C. T. Hatcher, chairman).—During the past year the members of the subcommittee have continued the work in conjunction with the members of Committee D-6 on Paper and Paper Products in the development of test procedures for determining pH values of insulating papers that will be satisfactory to both groups. With the exception of a few details the procedure has been agreed upon and it is expected that approval of the test will be obtained during the coming year.[10] Consideration is also being given to the question of obtaining a water extract conductivity test.

The Section on Physical Tests has been continuing its work on revising the test methods now appearing in Tentative Methods D 202 so as to have them in agreement with the same procedures of Committee D-6. In accordance with this procedure, either revised Tentative Methods D 202 or the methods of committee D-6 were adopted for the procedures covering sampling method, weight determination method, apparent density method, and rate of impregnation method. Work is also in progress on a new air resistance method.

The Section on Mechanical Strength is continuing its work with Committee D-6 on the revision of the tensile strength method.

Subcommittee IX on Mica Products (E. O. Hausmann, chairman).—The work on developing color photo transparencies of raw mica is being continued with the hope that satisfactory and acceptable standards may be developed.

Specifications for bonded mica are

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[10] See Editorial Note, p. 400.

under consideration. Some progress is being made on significance of tests.

An editorial change was made last year in Standard Methods of Testing Pasted Mica Used in Electrical Insulation (D 352 – 39) in which the test temperature was changed from "230 ± 5 C. (446 ± 9 F.)" to read "160 ± 5 C. (320 ± 9 F.)." The former test temperature was considered far too high for this material. This editorial revision was approved by letter ballot of Committee D-9 which consists of 118 members; 61 members returned their ballots, of whom 19 voted affirmatively, 0 negatively, and 42 members marked their ballots "not voting."

A very comprehensive round-robin and "Q" test on block mica has been completed. Seven laboratories participated. The purpose was three-fold: (*1*) to determine the degree of agreement between various laboratories using the rapid method, (*2*) to check the "Q" calibration of the meter used in the rapid test method, and (*3*) to determine if circular lead foil electrodes 2 in. in diameter, affixed with petrolatum were satisfactory for measuring the "Q" and dielectric constant of block mica approximately 0.020 in. in thickness at 1 megacycle.

Results of the round-robin test show: (*1*) good agreement between laboratories using either the rapid "Q" method or suitable bridges, (*2*) good agreement in calibration of the various "Q" instruments, and (*3*) satisfactory results in power factor "Q," and dielectric constant with use of 2-in. lead-foil electrodes.

On the basis of this round-robin test a paper entitled "Electrical Quality Classification of Raw Mica by a Rapid, Direct-Reading Method"[11] has been prepared by K. G. Coutlee, chairman of Section 3 of this subcommittee.

Subcommittee X on Conditioning (R. Burns, chairman).—Principal activity during the past year consisted of further revision of the Tentative Method for Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 45 T) to add a functional procedure B. This procedure requires that where the specific effects of exposure to severe atmospheric moisture are to be determined the specimens shall be subjected to 90 per cent relative humidity at 35 C. for four days.

Subcommittee XI on Significance of Tests (J. H. Adams, chairman).—Considerable attention was given to formulating a statement as to what shall commonly be the proper content for statements on significance of tests. Discussion was crystallized into tentative proposals which are under consideration. A revised statement as to the significance of resistivity tests was prepared and approved by letter ballot. Revision of statements as to the significance of dielectric strength and dielectric constant and power factor tests was initiated. Statements prepared by other subcommittees as to the utility of tests used, to the inspection of materials under their jurisdiction, were reviewed for any helpful advice which might be offered.

Subcommittee XII on Electrical Tests (Gordon Thompson, chairman).—Although there has been considerable activity during the year, the results shown by standard test methods are confined to revisions in Tentative Methods of Tests for Power Factor and Dielectric Constant (D 150 – 45 T) where a section has been added for the resonance rise method (familiarly known as the "Q-Meter" method), and in Standard Methods of Test for Insulation Resistance of Electrical Insulating Materials (D 257 – 45) where recognition has been given to the procedures called for by joint Army-Navy specifications. There has been some activity in the measurement of dielectric properties of materials at television and radar frequencies, and in tests for arc resistance.

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[11] See p. 1486.

Subcommittee XIII on Mechanical Tests (C. R. Stock, chairman).—Subcommittee XIII was assigned a new scope at the June, 1945, meeting in New York, N. Y., to read as follows:

> Prepare, where this work does not fall in the province of other standing committees, provide or advise concerning those methods of test for mechanical and thermal properties which are applicable to electrical insulating materials, such as elasticity, tensile strength, viscosity, thermal conductivity, etc., required by any of the other subcommittees; also coordinate and bring into uniformity so far as possible the various mechanical and thermal test methods for which Committee D-9 is responsible.

Five sections have been appointed to investigate and correlate the following types of tests:

1. Compression,
2. Dynamic (for example, impact),
3. Tension,
4. Flexure, and
5. Hardness.

Work has started on the first four of these tests along the same general lines; namely, the basic requirements for such test methods have been ascertained, some by means of questionnaires, and the deviations in detail of existing methods have been collected and tabulated. Further work will involve the establishment of reference requirements which will make for uniformity among test methods dealing with like properties.

The section on hardness is awaiting further developments in Committee D-20 on this subject.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, R. W. Orr.
Vice-Chairman, Gordon Thompson.
Secretary, W. A. Zinzow.

This report has been submitted to letter ballot of the committee, which consists of 118 voting members; 70 members returned their ballots, of whom 63 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

MYRON PARK DAVIS,
Chairman.

W. A. EVANS,
Vice-Chairman.

W. A. ZINZOW,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-9 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Methods of:
Testing Electrical Insulating Oils (D 117 - 46 T), and
Testing Askarels (D 901 - 46 T).

Revision of Tentative Methods of:
Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 - 46 T), and
Testing Vulcanized Fibre Used for Electrical Insulation (D 619 - 46 T).

These recommendations were accepted by the Standards Committee on November 23, 1946, and appear in the 1946 Book of A.S.T.M. Standards, Part III-B, under the designations indicated above.

On December 31, 1946, the Standards Committee accepted the recommendation of Committee D-9 that the proposed Methods of Testing Varnished Glass Fabrics and Varnished Glass Fabric Tapes be published as tentative. The new tentative methods have been assigned the designation D 902 - 46 T, and appear in the 1946 Book of A.S.T.M. Standards, Part III-B.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR ELECTRICAL INSULATING MATERIALS

In this Appendix are given recommendations affecting certain standards and tentatives covering electrical insulating materials which are referred to earlier in this report.[1] The standards and tentatives appear in their present form either in the 1944 Book of A.S.T.M. Standards, Part III, or the 1945 Supplement, Part III, as indicated by the final number in the A.S.T.M. designation.

New Tentatives

Tentative Methods of Test for Nonrigid Polyvinyl Tubing:

These methods are intended for test procedures to be used in evaluating nonrigid polyvinyl tubing or its copolymers with vinyl acetate for specification purposes. Most of the test methods described in this tentative have been developed for this purpose. Specifications covering these materials and using these methods are now being prepared. It is recommended that these methods, appended hereto,[2] be accepted for publication as tentative.

Tentative Method of Test for Dielectric Strength of Insulating Oil of Petroleum Origin:

This method of test describes procedures to be used in measuring the dielectric strength of insulating oils. It is the first of a series of test methods to be written up as separate tentatives which were formerly covered by the Standard Methods of Testing Electrical Insulating Oils (D 117 – 43). It is expected that when this series of tentatives are completed they will be referred to in Methods D 117 by title and designation only. This revision of methods D 117 has been under consideration for some time. It is recommended that this method, appended hereto,[2] be accepted for publication as tentative.

Tentative Method of Test for Inorganic Chlorides and Sulfates in Insulating Oils:

This is the second of the series of revisions of methods D 117 to be written up as a separate tentative. It describes in general the test procedure for inorganic salts covered by the test as now published in Methods D 117. A few minor changes to make the method more accurate have been incorporated. It is recommended that this method, appended hereto,[2] be accepted for publication as tentative.

Tentative Specifiations for Communication and Signal Pin-Type Lime-Glass Insulators:

These specifications were the former Emergency Specifications for Communication and Signal Pin-Type Lime-Glass Insulators (ES – 41), covering pin-type lime-glass insulators for use in supporting conductors on open-wire communication and signal lines. It is recommended that these specifications, with minor revisions, be accepted, for publication as tentative.[2]

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[1] See p. 393.

[2] These methods and specifications were accepted as tentative by the Society and appear in the 1946 Book of A.S.T.M. Standards, Part III-B

Revisions of Tentatives

Tentative Methods of Testing Molded Materials Used for Electrical Insulation (D 48 – 45 T):

This revision consists of the addition to these methods of an appendix giving brief descriptions of the utility of the test methods referred to, together with a reference section referring to these statements, which is to be inserted as Section 2 directly following the scope paragraph. The statements are intended to give a convenient indication of the utility of the information which may be gained by the performance of the tests. This revision, appended hereto,[3] does not involve any change in the manner in which the tests are conducted.

Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Materials (D 150 – 45 T):

This revision involves the addition of another method used for measuring power factors and dielectric constants of dielectrics. The method is commonly used and identified as the "Resonant Circuit, Resonance Rise Method."

Section 2 (d).—Add the following note to this section:

NOTE.—The storage factor or quality factor, Q, is the reciprocal of the dissipation factor, D.

New Section.—Add a new Section 29 to read as follows, renumbering the subsequent section accordingly:

29. *Resonant Circuit, Resonance Rise Method.*—(*a*) This method has been found suitable for frequencies from 50 kilocycles per second up to 10 megacycles per second. The method makes use of the fact that when a known radio-frequency voltage is introduced into a resonant circuit the ratio of this voltage to the voltage appearing across either the inductor or the capacitor of the resonant circuit is approximately the dissipation factor of the circuit. While this voltage can be introduced into the circuit by inductive, capacitive, or resistive coupling, its magnitude is more easily measured for resistive coupling, as shown in Fig. 15 (accompanying Fig. 1).

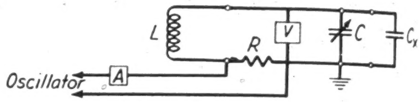

FIG. 1.—Resonant Circuit, Resonance Rise Method.

A radio-frequency oscillator furnishes to resistor R a constant current which is measured by radio-frequency ammeter A. This resistor shall be designed to have a very small reactance so that its impedance will differ from its resistance, at the highest operating frequency, by a negligible amount.[a]

The resonant circuit is composed of fixed inductor L and variable air capacitor C. From the vacuum tube voltmeter V is read the voltage developed across capacitor C. The dissipation factor of the entire circuit is the ratio of the voltage drop across resistor R due to current I from the oscillator, to the voltage E developed across capacitor C,

$$D_1 = \tan \delta = \frac{IR}{E} \quad \ldots\ldots\ldots(24)$$

(*b*) When all components required for this measurement (oscillator, ammeter A, resistor R, and vacuum-tube voltmeter V) are assembled in a single unit, the vacuum-tube voltmeter, V, can be calibrated directly in terms of dissipation factor D[b] by choosing a standard oscillator current I and always adjusting the current as indicated by ammeter A to this value.

(*c*) The vacuum-tube voltmeter, oscillator, and tuning capacitor shall in general conform to the requirements prescribed in Section 28 (*d*), (*e*), and (*f*).

(*d*) The specimen capacitor, with electrodes affixed, shall be connected by short low-resistance leads to the terminals of capacitor C. A suitable inductor L shall be selected which will permit resonance at the chosen frequency. The capacitance of capacitor C and the reading of the vacuum-tube voltmeter V at resonance shall be designated C_2 and D_2, respectively.

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[a] A commonly used value of resistance is 0.04 ohm for which the inductance can be reduced to a value of 0.07 mμh. The ratio of impedance to resistance at 10 megacycles is then 1.006.

[b] The voltmeter is frequently calibrated in terms of storage factor Q, which is the reciprocal of D, which provides an approximately uniform scale.

[3] These revised methods and specifications were accepted by the Society and appear in the 1946 Book of A.S.T.M. Standards, Part III-B.

The contact to the ungrounded electrode of the test specimen shall be broken by raising the connecting wire a small amount (about $\frac{1}{4}$ in. if the wire is bare and of small diameter). Resonance shall be reestablished by adjusting capacitor C, and the new readings of capacitor and voltmeter, C_1 and D_1, shall be noted.

Then:

$$D = \tan \delta = \frac{(D_2 - D_1)C_1}{C_1 - C_2} \quad \ldots\ldots (25)$$

$$C_p = C_1 - C_2 \quad \ldots\ldots\ldots (26)$$

where:

C_p = capacitance of the specimen, in micro-microfarads,

C_1 = capacitance of the standard air capacitor with the specimen out of circuit, in micro-microfarads,

C_2 = capacitance of the standard air capacitor with the specimen in circuit, in micro-microfarads,

D_1 = dissipation factor indicated by voltmeter with specimen disconnected, and

D_2 = dissipation factor indicated by voltmeter with specimen connected in parallel with standard capacitor.

NOTE.—When the voltmeter is calibrated in terms of storage factor, Q, Eq. 25 becomes:

$$D = \frac{1}{Q} = \frac{Q_1 - Q_2}{Q_1\,Q_2} \times \frac{C_1}{C_1 - C_2}$$

where:

Q_1 = storage factor indicated by voltmeter with sample disconnected, and

Q_2 = storage factor indicated by voltmeter with sample connected in parallel with standard capacitor.

Since this expression for dissipation factor of the specimen depends on the difference between two observations of dissipation factor, $(D_2 - D_1)$, the inductor L should be so chosen as to have as small a dissipation factor as possible. It should be possible to obtain inductors with dissipation factors not larger than 0.003 (storage factors not smaller than 330) in the frequency range for which this method is recommended.

Figures 15 and 16.—Renumber as Figs. 16 and 17 respectively.

Tentative Methods of Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 – 45 T):

This revision concerns the methods of sampling used in this procedure. A complete new statement of the method of sampling is presented more specifically on several points. It is the result of several years of experience with the present method of sampling.

Section 2.—Replace this section on sampling by the following:

2. *Sampling.*—(*a*) For batches consisting of rolls 15 in. or more in width, a sample at least 5 sq. ft. in area cut across the entire width of the roll shall be taken from every tenth roll of the batch, chosen in such a manner as to well represent the entire batch. In case the paper is available in rolls less than 15 in. in width, the length of the sample to be taken may be such that the area is less than 5 sq. ft. but shall be at least 4 ft. in length.

Where paper is in ribbon or pad form and in dowels; that is, a number of pads assembled on a dowel stick in the same order as slit from the parent roll, the sample should consist of specimens taken from the pads so that the entire width of the parent roll is represented. A minimum number of pads shall be sampled, the total width of which shall be equal to at least ten per cent of the width of each dowel. A minimum number of 4 pads from each dowel shall be sampled.

For batches in sheet form, sample sheets totaling at least 5 sq. ft. in area shall be taken from each tenth bundle or package chosen in such a manner as to well represent the entire batch.

In the case of either type of package no less than 5, nor more than 20 units shall be sampled, taken as follows:

Total Units in Shipment	No. of Units Selected
Less than 100	5
100 to 399	5 per cent
400 and over	20

NOTE.—It is recommended that in the case of paper in roll form, several turns be discarded before the samples are taken.

(*b*) The tests for physical properties shall be made on each sample insofar as the specimens are of sufficient width. If the samples are to be used for the determination of moisture in the batch, extreme care shall be taken to avoid change in moisture content in the samples before they are weighed as is provided for in the moisture method (Section 4).

Tentative Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (D 295 – 44 T):

These revisions involve the conditioning treatment required for this material prior to testing. The principal changes are those of conditioning at 50 per cent relative humidity instead of 60 to 65 per cent relative humidity, and making it possible to test the material in the as-received condition.

Section 4.—Replace this section on conditioning by the following:

4. *Conditioning.*—(*a*) If it is desired to test the material in the condition as received by the purchaser, the test samples shall not be specially conditioned, but the packages containing the rolls of cloth or tape from which the samples are to be taken shall be allowed to reach approximately test room temperature before the packages are opened and the samples cut. Specimens to be tested shall be removed from the roll as required and tested immediately, unless otherwise specified.

(*b*) Where it is desired to test in a controlled atmosphere, the test specimens shall be conditioned for 48 hr. in air maintained at a temperature between 20 to 30 C. (68 to 86 F.) and at a relative humidity of 50 ± 2 per cent.

(*c*) If a conditioning cabinet or chamber is used, test specimens shall be subjected to test immediately upon withdrawal from the cabinet or chamber, unless otherwise specified.

Section 40.—Replace this section on conditioning by the following:

40. *Conditioning.*—The test specimens shall be conditioned by one of the following two methods:

(*a*) When the greatest duplicability of results is desired, the test specimens in one thickness shall be heated to 105 ± 3 C. for 1 hr. without vacuum and then for 2 hr. at 106 ± 3 C. in a vacuum having an absolute pressure not exceeding 1 mm. of mercury.

(*b*) Where the vacuum treatment is not feasible or where tests in the as-received condition are desired, or where an approximation to some conditions of use is desired, the test specimens shall be conditioned in accordance with Section 4.

(*c*) In the case of dispute the vacuum treatment shall be considered the referee method.

Tentative Method for Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 45 T):

This revision involves a change in tolerance for test temperatures of –25 C. or below and the addition of a second functional procedure with clarifying explanations to serve as a guide to the choice of procedures to be used. The revised method is appended hereto.[3]

Tentative Specifications for Laminated Thermosetting Materials (D 709 - 44 T):

These revisions cover certain changes in property values, and the addition of several new tables of dimensional tolerances on large round tubing and on rectangular tubing. These changes have been approved by the National Electrical Manufacturers Association, and A.S.T.M. Committee D-20 on Plastics. The revised specifications are appended hereto.[3]

Tentative Specifications for Low and Medium Voltage Pin-Type, Lime-Glass Insulators (D 730 – 43 T):

The present Tentative Specifications D 730 include many methods used in testing. This revision involves the deletion of these test methods from the specification and substitution for them of references to the same methods of test which are covered in the revised Standard Methods of Testing Pin-Type, Lime-Glass Insulators (D 468 – 46) which are discussed later in this report. It is a clarifying revision designed to make D 730 a specification and not a method of test and specification combined.

Sections 12 to 20.—Replace the methods of test in these sections by the following new Sections 12 to 15:

12. *Gaging.*—A gage of the design and dimensions shown in Fig. 1 of the Standard Methods of Testing Pin-Type Lime-Glass Insulators (A.S.T.M. Designation: D 468) shall be screwed tightly into the insulator pin cavity and the dome clearance (distance between line "A" of the

gage drawing and the crown of the pin cavity) shall be measured. There shall be no perceptible play or rocking of the insulator when seated on the gage. The gage shall then be removed from the insulator, counting the number of turns to disengage. All insulators which have been gaged shall be discarded.

13. *Thermal Shock Test.*—The specimen insulators shall be subjected to the stipulated thermal shock test (manual method or machine method) as described in Section 5 of A.S.T.M. Methods D 468. Tested insulators shall be discarded.

14. *Mechanical Strength Test.*—The specimen insulators shall be subjected to the mechanical strength test as described in Section 7 of A.S.T.M. Methods D 468.

15. *Electrical Voltage Tests.*—The specimen insulators shall be subjected to the specified dry-flashover, wet-flashover, and critical impulse-flashover tests as described in Sections 8 to 13 of A.S.T.M. Methods D 468.

As part of the general program for revising specifications D 730 and methods D 468 it is planned to revise the Emergency Specifications for Communication and Signal Pin-Type Lime-Glass Insulators (ES -41) and then recommend the specifications for publication as tentative. Final action on this proposal will be taken by Committee D-9 at its June meeting. The following contemplated changes are shown here for record purposes only:

Section 5.—Change Paragraph (*b*) to read as follows:

(*b*) *Screw Threads.*—The insulators shall have the standard screw threads to fit the wooden pin gage or the CSC gage, as applicable. The dimensions and construction of these gages shall be as described in Section 6 and in Figs. 2 and 3, respectively, of the Standard Methods of Testing Pin-Type, Lime-Glass Insulators (A.S.T.M. Designation: D 468). The thread shall be smooth, of uniform pitch and well centered in the insulator.

In Paragraph (*c*) on gage fit delete the reference to "(Fig. 2)" and "(Fig. 3)" from item (1).

Section 11.—Change this section on thermal shock test to read as follows: "11. The specimen insulators shall be subjected to the stipulated thermal shock test (manual method or machine method) as described in Section 5 of A.S.T.M. Methods D 468."

Figures 2 and 3.—Delete these figures showing gages for wooden pin-type and carrier current insulators.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

Standard Methods of Test for Insulation Resistance of Electrical Insulating Materials (D 257 – 45):

The first of two revisions of this method consists of a group of definitions of five terms used about the accuracy of which there was some question and a certain amount of discrepancy as compared with other statements found elsewhere in the methods.

The second of these revisions consists of changes in the method for measuring insulation resistance under certain specific conditions. It is largely a revision of the method to bring the A.S.T.M. and the Joint Army-Navy procedures into line with each other. These revisions were arrived at at a conference between members of A.S.T.M. Committees D-9 and D-20 and members from Army and Navy laboratories.

Section 2.—Replace the present definitions in this section by the following:

2. *Definitions:* (*a*) *Volume Resistance.*—The volume resistance between two electrodes which are in contact with, or embedded in a specimen, is the ratio of the d-c. voltage applied to the electrodes, to that portion of the current between them that is distributed through the volume of the specimen.

(*b*) *Volume Resistivity.*—The volume resistivity of a material is the ratio of the potential gradient in volts per centimeter parallel to the current flow in the material, to the current density in amperes per square centimeter.

NOTE 1.—Volume resistivity of a material is numerically equal to the volume resistance when measured between two electrodes which cover opposite faces of a centimeter cube of the material.

(*c*) *Surface Resistance.*—The surface resistance between two electrodes which are on the surface of a specimen is the ratio of the d-c. voltage applied to the electrodes to that portion of the current between them which is in a thin layer of moisture or other semi-conducting material that may be deposited on the surface.

(*d*) *Surface Resistivity.*—The surface resistivity of a material is the ratio of the potential gradient in volts per unit distance parallel to the current flow along its surface, to the current in amperes per unit width of surface.

NOTE 2.—Surface resistivity of a material is numerically equal to the surface resistance between two electrodes forming opposite sides of a square. The size of the square is immaterial.

(*e*) *Insulation Resistance.*—The insulation resistance between two electrodes which are in contact with, or embedded in a specimen, is the ratio of the d-c. voltage across the electrodes to the total current between them. It is dependent upon both the volume and surface resistivities of the material as well as upon the configuration of the electrodes.

Section 3.—Change this section on test specimen to read as follows by the addition of the italicized words: "The test specimen for solid insulating materials shall be either in the form of a flat plate, *a cylinder*, or a tube."

Section 4.—Add the following new paragraph at the end of this section on electrodes:

For insulation resistance measurements on molded or laminated materials the electrodes shown in Fig. 3 (tapered pins) shall be used, unless otherwise agreed upon by the manufacturer and the purchaser. Figures 1 and 2 show electrodes which are in common use and which may be specified in place of the tapered pins, upon mutual agreement between the manufacturer and the purchaser.

Figure 3.—Change the dimensions of this figure to show a block 2 by 3 in., thickness not specified. The holes shall be $\frac{3}{16}$ in. on 1 in.-centers, reamed to fit Pratt and Whitney No. 3 tapered pins. Holes shall be 1 in. in from the sides and the ends of the block.

Section 11.—Change this section on conditioning to read as follows:

11. (*a*) Unless otherwise agreed upon, determinations of insulation resistance of solid dielectrics shall be made on specimens which have been exposed for a period of 96 hr. in an atmosphere maintained at a relative humidity of 90 ± 2 per cent and a temperature of 35 ± 1 C. (95 ± 2 F.). When introduced into the conditioning chamber the specimen shall be at a temperature of 35 C. within + 5 or − 0 C. Measurement of insulation resistance shall be made while the specimen is in the conditioning chamber maintained at the above specified relative humidity and temperature. An alternative conditioning procedure[4], requires that the determination of insulation resistance of solid dielectrics shall be made on specimens which have been immersed in water at 50 C. (122 F.) for a period of 96 hr., then cooled in water and wiped off. Other conditioning processes may be used if agreed upon by the manufacturer and the purchaser.

Where electrode leads pass through the walls of the chamber, care shall be taken to insure against current leakage at these points by suitable guarding or by passing leads through bushings of wax or other nonwetting materials.

(*b*) *Humidity and Temperature Control.*—Any method of humidity and temperature control may be used provided the stipulated limits of plus or minus 2 per cent for humidity and plus or minus 1 C. (2 F.) for temperature are not exceeded. These limits are based on the capabilities of commercial conditioning equipments and, while generally acceptable for acceptance testing, are not close enough for referee work. In the latter case, humidity should be maintained at plus or minus 1 per cent and temperature at plus or minus 0.5 C. (1 F.) or at closer limits, if obtainable, as agreed upon by the manufacturer and the purchaser. In small chambers, such as desiccator jars using acid or salt solutions, a small leak should be provided to maintain the conditioned space at approximately atmospheric pressure. In such containers a sulfuric acid-water mixture may be used to maintain the humidity at any desired point (see Fig. 6). Saturated solutions of certain salts such as sodium chloride, potassium chloride, and sodium nitrate may also be used to maintain definite humidity conditions. The temperature of the desiccator or other container shall be maintained constant, and an adequate surface exposure of the mixture or solution shall be provided. A gentle air circulation should be maintained, but not sufficient to pick up droplets of the solution.

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[4] This procedure is specified in the Joint Army-Navy Specifications.

Section 16.—Change the sentence following Eq. 6 to read: "In the case of metal strips applied to a thin tape or strip of insulating material the surface resistance per square may be calculated by the following formula:"

Change the legend for "*b*" following Eq. 7 to read: "b = the average width of the tape or strip of insulating material, and".

Standard Methods of Testing Pin-Type, Lime-Glass Insulators (D 468 - 42):

This revision is largely editorial in nature involving two changes. The first of these covers the thermal shock test. For nearly three years Methods D 468 have carried an appendix (Appendix IV) as a tentative revision of these methods, describing the machine method of making the thermal shock test. The text of the Appendix differed from that in the methods in only a few characteristic sentences. These sentences are now to be incorporated in the revised methods. The machine method differs from the manual method in no significant detail and now takes its place as a fully optional procedure.

The second change involves the transfer to methods D 468 of the detailed description of the methods for carrying out many of the tests which were formerly included in Tentative Specifications D 730 - 43 T as outlined above under Specifications D 730.

Standard Method of Test for Acetone Extraction of Phenolic Molded or Laminated Products (D 494 - 41):

Committee D-9 joins with Committee D-20 on Plastics in recommending the proposed revision for a new Section 2 on significance of test as covered in the Appendix to the report of Committee D-20.[5]

REVISION OF STANDARD AND REVERSION TO TENTATIVE

Standard Methods of Testing Varnishes Used for Electrical Insulation (D 115 - 41):

The revision of this method is rather complete but largely editorial in nature and does not change any of the tests involved in any essential way. The proposed revised methods, appended hereto,[3] are recommended for publication as tentative as further changes are contemplated.

ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that the Tentative Method of Test for Power Factor and Dielectric Constant of Natural Mica (D 351 - 42 T) be approved for reference to letter ballot of the Society for adoption as standard with revisions as given below. If this action is approved, this method will then be combined with the Standard Methods of Test for Grading and Classification of Natural Mica (D 351 - 38).

Section 1.—At the end of the first sentence delete the words "and mica splittings" and substitute the following: "0.007 to 0.030 in. in thickness and mica films or capacitor splits 0.0008 to 0.004 in. in thickness."

Section 2 (a).—At the end of the sentence, add the following: "for either block mica or films. Greater precautions will be necessary in the use of either method in testing mica films thinner than 0.002 in. due to greater sensitivity to contamination and humidity effects."

Add the following new Paragraphs *(d)* and *(e)* to this section:

(*d*) *Lead-Foil Electrodes.*—The use of lead foil electrodes 0.0005 in. in thickness and 2.0 in. in diameter is satisfactory for block mica 0.015 to 0.030 in. in thickness.

(*e*) *Rapid, Direct-Reading Method.*—Where classification of relative order of magnitude of power factor or its reciprocal *Q* value of block mica or films is desired the rapid, direct-reading method specified in the Tentative Specifications for Natural Block Mica and Mica Films Suitable for Use in Fixed Mica-Dielectric Capacitors (A.S.T.M. Designation: D 748) is satisfactory.

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[5] See p. 461.

Section 3 (a).—Add the following at the end of the first sentence: "When using steel electrodes or mercury electrodes (Section 6 *(b)* or *(c)*). Only one test specimen shall be tested at a time with lead-foil electrodes (Section 6 *(c)*). When the rapid, direct-reading method is used, test specimens shall conform to the requirements specified in A.S.T.M. Specifications D 748."

Section 4.—Letter the first paragraph as *(a)*. Reletter Paragraph *(a)* as *(b)* and change the first sentence to read as follows: "The surfaces of the specimen with the exception of the specimens used in Section 6 *(e)* shall be thoroughly and carefully cleaned with a camels hair brush dipped in petrolic ether or vapor degreased using trichlorethelene."

Section 5 (a).—Add the following clause to the beginning of the first sentence: "When steel electrodes, mercury electrodes, or lead-foil electrodes (Section 6 *(b)*, *(c)* or *(d)* are used."

Add the following as a second sentence to this paragraph: "When the rapid, direct-reading method (Section 6 *(e)*) is used, the power factor or *Q* value shall be determined in accordance with the procedure described in A.S.T.M. Specifications D 748."

Section 5 (b).—In the third line of this paragraph after the word "electrodes" add the words "specified in Section 2*(b)*."

Section 5 (c).—In the first line of this paragraph after the word "electrodes" add the words "specified in Section 2 *(c)* and lead-foil electrodes specified in Section 2 *(d)*."

Section 6.—In the first sentence after the word "measurement" add the following: "when using steel electrodes or mercury electrodes."

ADOPTION AS STANDARD OF TENTATIVE REVISIONS OF STANDARDS

Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 – 43):

The committee recommends that the tentative revision[6] of these methods, involving changes in Sections 5 to 9, 14, 30*(b)*, 33*(d)*, and 51 submitted in June, 1943, and modified in August, 1943, and June, 1944, be approved. These revisions refer to the tests for tensile strength, compressive strength, dielectric strength, and co-efficient of thermal expansion.

Standard Methods of Testing Laminated Tubes Used in Electrical Insulation (D 348 – 42):

The committee recommends that the tentative revision[6] of these methods, referring to dimensional measurements, and dielectric strength measurements submitted in June, 1942, and modified in June, 1943, be approved.

Standard Methods of Testing Laminated Round Rods Used in Electrical Insulation (D 349 – 42):

The committee recommends that the tentative revisions[6] of these methods, as submitted in June, 1942, modified in June, 1943, and issued in June, 1944, and which refer to a procedure for dielectric strength be approved.

Standard Methods of Test for Grading and Classification of Natural Mica (D 351 – 38):

The committee recommends that the tentative revisions[6] of these methods, issued in June, 1942, and June, 1944, which refer to the addition of a new quality classification table and authorization for combining Tentative Methods D 351 – 42 T with Standard Methods D 351 – 38, be approved. In effecting this consolidation of methods a number of editorial changes will be necessary.

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[6] 1945 Supplement to Book of A.S.T.M. Standards, Part III.

REPORT OF COMMITTEE D-10*

ON

SHIPPING CONTAINERS

Committee D-10 on Shipping Containers held one meeting during the year at Atlantic City, N. J., April 5, 1946. There were 43 members and visitors in attendance. Two visitors were from the Industrial Research Council of The Netherlands, two from the Army-Navy Packaging Board, one from the Bureau of Supplies and Accounts, U. S. Navy, and one from the Chicago Quartermaster Depot. In addition, the chairman read a letter from the Utilization Officer, Forest Research Institute, New Forest, India, asking to be kept informed of the activities and discussions of the committee. This wide-spread interest is a clear indication of the importance attached to specifications and test methods for shipping containers.

During the past year the committee has continued its growth, and now consists of 70 members of whom 30 are classified as producers, 24 as consumers, and 16 are general interest members.

The officers of Committee D-6 on Paper and Paper Products, representatives from Society headquarters, and the officers of Committee D-10 met in July, 1945, to discuss the scope of each committee and eliminate overlapping activities. As a result of this meeting, the Executive Committee of the Society placed the sole responsibility for the development of shipping container test methods and specifications with Committee D-10. R. L. Beach was appointed by Committee D-6 as its representative on Committee D-10. E. Dahill represents Committee D-10 on Committee D-6.

Subsequent to the 1945 Annual Meeting, Committee D-10 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Methods for:

Compression Test for Shipping Containers (D 642 – 45 T) to supersede Standard Method D 642 – 43, and

Drop Test for Shipping Containers (D 775 – 45 T) to supersede Tentative Method D 775 – 44 T.

Methods D 642 and D 775 were formerly under the jurisdiction of Committee D-6 and were transferred to Committee D-10 in accordance with the arrangements mentioned earlier in this report. These recommendations were accepted[1] by the Committee on Standards on August 27, 1945, and the tentative methods appear in the 1945 Supplement to Book of A.S.T.M. Standards, Part III.

On February 15, 1946, the Administrative Committee on Standards accepted[1] revisions in the Tentative Method of Drum Test for Containers in Small Revolving Hexagonal Drum Box-Testing Machine (D 782 – 44 T). This method, before revision, had also been under the jurisdiction of Committee D-6. The revised method will appear in the 1946 Book of A.S.T.M. Standards, Part III-B.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] In submitting these recommendations to the Administrative Committee on Standards, Committee D-10 reported the following results of the letter ballot vote from a committee membership of 70: D 642 – 45 T, affirmative 44, negative 1, ballots marked "not voting" 7; D 775 – 45 T, affirmative 45, negative 2, ballots marked "not voting" 5; D 782 – 44 T, affirmative 52, negative 3, ballots marked "not voting" 9.

A vibration test for shipping containers was submitted to letter ballot of Committee D-10 but it received several negative votes and was referred back to the subcommittee. It is now being held awaiting more definite developments in regard to the magnitude and frequency of the forces acting on shipping containers in transportation.

A list of definitions was prepared and submitted to the members of Committee D-10 after approval by Subcommittee I. There were many suggestions for changes and additions and the list is now in process of revision by the subcommittee.

During the year the secretary of Committee D-10 sent a questionnaire to every member so that the committee officers would have authentic information on each member in regard to:

Company relation to shipping container problems,

Individual's activity in shipping containers,

Primary interest in shipping containers,

Testing equipment available, and

Preference for subcommittee service.

The answers received have been very helpful in setting up and revising the subcommittees.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, T. A. Carlson.
Vice-Chairman, R. L. Beach.
Secretary, E. R. Stivers.
Members of Advisory Committee: Edward Dahill and P. P. Kennedy

NEW TENTATIVE METHOD

The committee recommends that the Method of Incline-Impact Test for Shipping Containers[2] be accepted for publication as tentative.

This recommendation has been submitted to letter ballot of the committee which consists of 70 members; 49 members returned their ballots, of whom 45 have voted affirmatively, 1 negatively, and 3 members marked their ballots "not voting."

ACTIVITIES OF SUBCOMMITTEES

Most of the subcommittees have been active throughout the year carrying on their work by correspondence. All but one of the five subcommittees met for an afternoon session on April 4, 1946, at Atlantic City, N. J. At this time they gave attention to work in progress and outlined a program for the future. For the coming year, the subcommittees have appointed the following chairmen:

Subcommittee I on Definitions of Terms, Edward Dahill, chairman,
Subcommittee II on Methods of Testing, E. R. Stivers, chairman,
Subcommittee III on Moisture and Water Vapor Resistance, R. W. Lahey,
Subcommittee IV on Performance Standards J. H. Toulouse,
Subcommittee V on Correlation of Tests and Test Results, R. C. McKee, and
Subcommittee VI on Interior Packing, W. B. Lincoln, Jr., chairman.

Some of the subcommittees have organized sections to increase their effectiveness.

This report has been submitted to letter ballot of the committee, which consists of 70 members; 52 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

EDWARD DAHILL,
Chairman.

E. R. STIVERS,
Secretary.

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[2] This method has been accepted as tentative by the Society and appears in the 1946 Book of A.S.T.M. Standards, Part III-B.

REPORT OF COMMITTEE D-11*

ON

RUBBER AND RUBBER-LIKE MATERIALS

Committee D-11 on Rubber and Rubber-Like Materials held one meeting during the year, in Pittsburgh, Pa. on February 28, 1946, in connection with the spring group meetings of A.S.T.M. committees. Several of the subcommittees have met separately during the year and considerable work has been carried on by correspondence.

Several changes have occurred in the representation of the committee in other organizations. A. M. Finley was appointed as the representative on ASA Sectional Committee L3 on Rubber-Lined Fire Hose replacing W. L. Sturtevant who retired from active membership on the committee after many years of service. Mr. Sturtevant has been appointed a consulting member on Committee D-11. J. H. Ingmanson was appointed to represent the committee on ASA Sectional Committee C59 on Electrical Insulating Materials following the resignation of E. D. Youmans. L. V. Cooper is replacing J. J. Allen as the committee's representative in the Section on Indentation Hardness of the Society's Committee E-1 on Methods of Testing.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1945 Annual Meeting, Committee D-11 presented to the Society through the Administrative Committee on Standards a number of recommendations which are listed in Table I, together with the results of the letter ballot vote in Committee D-11 and the date of acceptance by the Standards Committee. The new and revised tentatives have been given the designations indicated and together with the tentative revision appear in the 1946 compilation of "A.S.T.M. Standards on Rubber Products."

The new specifications D 866 for GR-S sheath compound are identical with those formerly published as Emergency Specifications (ES-6a) except that the scope was modified to limit service applications of this material to temperatures of minus 25 C. and higher and a requirement was added specifying a minimum elongation of 200 per cent after 48 hr. in the oxygen pressure aging test. These changes were considered necessary to assure proper service application and performance. The tentative revision of Standard Specifications D 469 covers the substitution of a new Table III on insulation resistance based on a value of *K* equal to 10,560 instead of the present table based on a value of *K* equal to 15,840. The new table is considered to be more satisfactory and in line with the present practice for this type of material.

The revisions in the Tentative Specifications D 754 and D 755 include a low-temperature restriction of minus 35 C. below which use of these insulating materials is not recommended and a change in the duration of the high-voltage test from 1 min. to a time interval of 5 min. The temperature limitation is intended to assure proper service application of the material and the increased time in the voltage test is a more severe requirement assuring better insulation qualities. The revisions in the cellular rubber specifications D 798 were extensive and detailed replacing all of the present tables in the specifications with new ones and rearranging the former permitted tolerances. These changes were the result of the experience of members of the committee in use of the specifications since their adoption two years previously and were considered necessary to represent usual commercial practice and to make these specifications more usable in the trade. In the immersion test Methods D 471, experience had indicated that the aniline points of the petroleum-base test oils were not controlled sufficiently close to assure reproducible results. This was corrected in the revisions recommended and in addition, standard sources of oils were established which would provide a means whereby oil held to closer tolerances than the ordinary commercial grades could be obtained. The revisions in Methods D 552 for testing cellular rubber products were relatively minor clarifying details concerning test equipment required.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

TABLE I.—RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS INCLUDING RECORD OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
New Tentative Specifications for:			
GR-S Synthetic Rubber Sheath Compound for Electrical Insulated Cords and Cables (D 866 - 46 T), *accepted January 10, 1946*	56	2	46
New Tentative Method for:			
Heat Aging of Vulcanized Natural or Synthetic Rubber by Test Tube Method (D 865 - 46 T) *accepted February 15, 1946*	69	1	18
Revision of Tentative Specifications for:			
Insulated Wire and Cable: Heat-Resisting Synthetic Rubber Compound (D 754 - 46 T), *accepted January 10, 1946*	60	0	44
Insulated Wire and Cable: Performance Synthetic Rubber Compound (D 755 - 46 T), *accepted January 10, 1946*	60	0	44
Cellular Rubber Products (D 798 - 46 T), *accepted January 10, 1946*	45	0	59
Revision of Tentative Methods of			
Test for Changes in Properties of Rubber and Rubber-Like Materials in Liquids (D 471 - 46 T), *accepted January 10, 1946*	65	0	39
Testing Cellular Rubber Products (D 552 - 46 T), *accepted January 10, 1946*	48	0	56
Tentative Revision of Specifications for:			
Insulated Wire and Cable: Heat Resisting Rubber Compound (D 469 - 41), *accepted January 10, 1946*	57	0	47

The new Test D 865 for heat aging of rubber by the test tube method was developed by Section IV of Technical Committee A on Automotive Rubber in connection with work looking toward improvement in the means for evaluating the resistance of synthetic rubbers to deterioration at high temperatures. Experience with existing methods had shown considerable variation and quite a number of attempts had been made to remedy this situation. The work of the section, which was published in the ASTM BULLETIN,[1] indicated that the proposed test tube technique offered a more satisfactory test procedure. Further study confirmed this and the committee decided to publish the method in order that additional confirmation and experience can be obtained.

EMERGENCY ALTERNATE PROVISIONS AND EMERGENCY SPECIFICATIONS

Emergency Alternate Provisions EA - D 353, EA - D 469a, and EA - D 532a were withdrawn by recommendation of Committee D-11 and acceptance of the recommendation by the Standards Committee on January 10, 1946, since the need for these provisions was no longer considered to exist. The emergency alternate provisions for the Standard Specifications for Friction Tape (D 69 - 38) and for Rubber Insulating Tape (D 119 - 38) are herein recommended for withdrawal and the portions of these provisions which are still essential are being incorporated in the standard specifications which will be returned to the tentative status. The committee will then have under its jurisdiction no emergency specifications and it will still have emergency alternate provisions in four specifications and one method of test, all of which deal with insulated wire and cable. The Subcommittee on Insulated Wire and Cable has appointed a section which is now working on the elimination of these remaining emergency alternate provisions and appropriate action to this end is expected to be taken through the Standards Committee immediately after the 1946 Annual Meeting.

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[1] ASTM Bulletin, Am. Soc. Testing Mats., No. 138, January, 1946, p. 33.

I. REVISIONS OF TENTATIVES

The committee recommends that the following eleven tentatives be revised as indicated below and continued as tentative:

Tentative Methods of Test for Compression Set of Vulcanized Rubber (**D 395 - 40 T**).[2]—Under method B, compression set under constant deflection, the percentage of compression employed in the test depends upon the hardness of the material being tested and is specified in a table given in Section 11 (*b*). This procedure is widely used in the automotive industry where it originated and Technical Committee A on Automotive Rubber has recommended a somewhat different and more specific table for the same purpose as given in Section 8 (*e*) of the Tentative Specifications for Rubber and Synthetic Rubber Compounds for Automotive and Aeronautical Applications (D 735 - 43 T). The committee has reviewed these two tables and concluded that there should be only one table and that the latter is the better.

The committee therefore recommends the following revision in Methods D 395:

Section 11 (*b*).—Delete the present table and substitute the following:

Durometer Hardness Number	Deflection, per cent of original thickness
30 ± 5	40
40 ± 5	40
50 ± 5	30
60 ± 5	30
70 ± 5	25
80 ± 5	25
90 ± 5	20

Tentative Methods of Testing Cellular Rubber Products (**D 552 - 46 T**).[3]—Work in the committee has indicated that the use of the specified dial gage for the smaller thicknesses is a more accurate means of measurement of thickness than a sliding caliper gage as previously required. The committee therefore recommends the following revisions:

Section 4.—Delete the last three sentences and substitute the following sentence: "The thickness shall be measured using the dial micrometer gage described in Section 6 (*b*)."

Section 6 (*b*).—Change to read as follows:

> (*b*) Thicknesses up to and including 1 in. shall be measured using a dial-type gage[a] having a maximum stem and foot weight of 20 g. and a foot 1¼ in. in diameter. Thicknesses over 1 in. shall be measured using a sliding caliper gage or as specified in Paragraph (*a*). When a sliding caliper gage is employed, the gage setting shall be made with the gage out of contact with the cellular rubber. The sample shall be passed through the previously set gage and the proper setting shall be the one when the measured faces of the gage contact the surfaces of the article without compressing it.

Section 6 (*c*).—Change to read as follows: "(*c*) The type of steel scale tape or dial gage and the graduations employed shall be so chosen as to give accurate measurements according to the specified permissible variations."

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[2] 1944 Book of A.S.T.M. Standards, Part III.

[a] A gage similar to Federal Products Co. No. D-81-S is satisfactory.

[3] Compilation of "A.S.T.M. Standards on Rubber Products," March, 1946.

Section 14.—Delete the references in the first sentence to compression-deflection value of less than 13 psi. and to 25 per cent compression. The sentence will then read as follows: "The apparatus and procedure shall be the same as prescribed in Method B of the Tentative Methods of Test for Compression Set of Vulcanized Rubber (A.S.T.M. Designation: D 395) except as follows: Test specimens shall be compressed 50 per cent of their original thickness."

This change is in line with the deletion of the requirements in the Tentative Specifications for Cellular Rubber Products (D 798) for compression set using 25 per cent deflection which is also being recommended.

Tentative Methods of Testing Asphalt Composition Battery Containers **(D 639 – 43 T)**:[2]

Section 13.—Change to read as follows:

13. *Apparatus:* (*a*) *Testing Machine.*—Tensile strength and elongation shall be determined on a power-driven apparatus of either the beam-weighing or pendulum type conforming to the following requirements:

(1) The applied tension as indicated by the dial or scale shall be accurate within 1 per cent.

(2) The indicator shall remain at the point of maximum load after rupture of the test specimen.

(3) The rate of travel of the power actuated grip shall be such that the average rate of load application shall be not more than 3.1 nor less than 2.9 lb. per sec. when measured with a non-extensible steel bar locked in the grips.

(4) Testing machines with a maximum capacity of 500 to 1500 lb. may be used to meet the conditions outlined above.

NOTE.—To establish a specification standard, it is advisable to specify the type of testing machine which is to be used to give the required tensile strength and elongation.

(*b*) *Grips.*—The grips shall be as described in Section 7 (*b*) of the Tentative Methods of Testing Hard Rubber Products (A.S.T.M. Designation: D 530).

New Section.—Add the following as a new Section 14, renumbering the subsequent sections accordingly:

14. *Calibration of Testing Machine.*—The testing machine shall be calibrated as described in Section 8 of the Tentative Methods of Testing Hard Rubber Products (A.S.T.M. Designation: D 530).

This change is necessary in order to provide for testing at a specified rate of load application rather than at a constant rate of jaw separation. The Section on Asphalt Composition Battery Containers of Subcommittee XXIII has done a large amount of work in trying to eliminate confusion and to improve reproducibility in testing for tensile strength and elongation. Several round-robin test programs have been carried out in six cooperating laboratories and the committee has shown that the rate of loading has a distinct effect upon the breaking strength but little or no effect upon elongation, low rates of loading giving lower tensile strength figures than higher rates of loading. The conclusion has also been reached that where different types of testing machines are in use it is more important to specify the rate of loading than the speed of jaw separation which is just one factor in the rate of loading.

Tentative Method of Test for Indentation of Rubber by Means of the Durometer **(D 676 – 44 T)**:[2]

Section 4 (*b*).—Change item (*1*) to read as follows: "(*1*) The durometer and the specimen shall be conditioned in air or in a mixture of air and carbon dioxide at the prescribed temperature for at least 30 min. before test."

This change permits the use of dry ice cold boxes and is in line with current practice.

Tentative Specifications for Rubber and Synthetic Rubber Compounds for Automotive and Aeronautical Applications **(D 735 – 43 T)**:[2]

Section 3 (*b*).—In the paragraph dealing with *Suffix Letters* insert the following sentences just prior to the listing of the significance of approved suffix letters:

In those cases where a specific test for the property covered by a suffix letter has not been made a part of these specifications, the suffix letter is not to be used until suitable tests have been arranged by agreement between the manufacturer and the purchaser. The manufacturer will not be held accountable for special tests worked out by the purchaser unless the tests are specified on part drawings or the manufacturer has been notified in writing regarding these tests previous to entering into a contract to furnish these materials.

Also, add the following approved suffix letters:

Suffix Letter	
G	Resistance to Tear required. Test to be arranged between the manufacturer and the purchaser.
H	Resistance to severe flexing required. Test to be arranged between the manufacturer and the purchaser.
J	Resistance to abrasion required. Test to be arranged between the manufacturer and the purchaser.
K	Adhesion to metal required. Natural and synthetic rubber compounds can be bonded to most metals. The bond obtained is dependent on many factors, such as: (*a*) Type of compound. (*b*) Type of metal. (*c*) Surface preparation of the metal. (*d*) Type of adhesive used. (*e*) Size and shape of the product. (*f*) Method of vulcanization. Uniform adhesion values should range upward from approximately 200 psi. for compounds of less than 50 durometer hardness, and from approximately 250 psi. for compounds of over 50 durometer hardness, provided the compounds are designed for use in bonding to metal. Standard Method of Test for Adhesion of Vulcanized Rubber to Metal (A.S.T.M. Designation: D 429) should be used when possible. However, it is usually desirable to run tests on the actual production parts, in which case, the adhesion value is measured by a tension pull normal to the plane of the metal surfaces and calculations are based on the projected area of the smaller metal part. It is recommended that the respective purchaser and manufacturer of products requiring adhesion to metal mutually agree on the test method and adhesion value required for each specific application.
L	Resistance to water absorption required. Test to be arranged between the manufacturer and the purchaser.
M	Inflammability resistance required. Test to be arranged between the manufacturer and the purchaser.
N	Resistance to impact required. Test to be arranged between the manufacturer and the purchaser.
P	Test for the degree of staining of surface finishes required. Methods of test and limits to be arranged between the manufacturer and the purchaser.
Z	Special tests of either the usual laboratory type or performance type required. Such tests to be arranged between the manufacturer and the purchaser.

Table II.—For the class RS compounds add the following A.S.T.M. hardness numbers and values for 20 per cent load deflection in the appropriate columns:

Grade Numbers	A.S.T.M. Hardness Number
RS300 to RS320–7, incl.	175 ± 25
RS400 to RS425, incl.	125 ± 25
RS500 to RS520, incl.	90 ± 20
RS600 to RS625, incl.	63 ± 17
RS700 to RS725, incl.	42 ± 12
RS804 to RS825, incl.	30 ± 10
RS907 to RS915, incl.	15 ± 10

Grade Numbers	Suffix D 20 per cent Load Deflection, psi.
RS315 to RS320–7, incl.	70 ± 15
RS415 to RS425, incl.	102.5 ± 22.5
RS515 to RS520, incl.	145 ± 30
RS615 to RS625, incl.	205 ± 45
RS715 to RS725, incl.	302.5 ± 72.5
RS820 to RS825, incl.	472.5 ± 102.5

Note.—Load deflection values not provided for compounds having a tensile strength of less than 1500 psi. nor for compounds over 80 Durometer hardness.

Table V.—Change the values for maximum compression set under Suffix B to read as given in the accompanying Table II:

These changes are the result of considerable work by Section IV of Technical Committee A and have been thoroughly studied and approved both by the section and by letter ballot of the entire Technical Committee. It is believed by those concerned that they will improve the specifications and increase their utility.

Tentative Method of Test for Low-Temperature Brittleness of Rubber and Rubber-Like Materials (**D 736 – 43 T**):[2]

Section 4.—Change the second sentence to read as follows: "With the plates in the open position separated 2½ in., the fixture containing the specimens shall be placed in the cold chamber and exposed for the specified period to cold, dry air or a mixture of air and carbon dioxide at the specified temperature."

TABLE II.

Grade No.	22 hr. at 158 F.	70 hr. at 212 F.
SC 305	70	...
SC 310	70	...
SC 315	70	...
SC 320	70	...
SC 400	...	75
SC 405	65	...
SC 415	65	...
SC 420	60	...
SC 425	60	...
SC 500	...	70
SC 507	60	...
SC 512	60	...
SC 520	50	...
SC 525	50	...
SC 530	50	...
SC 600	...	70
SC 608	55	...
SC 614	55	...
SC 620	50	...
SC 625	50	...
SC 630	45	...
SC 700	...	65
SC 707	50	...
SC 717	45	...
SC 725	40	...
SC 800	...	65
SC 804	50	...
SC 808	50	...
SC 815	45	...
SC 902	50	...
SC 910	50	...

Make the same change in the second sentence of Section 2 (*a*) adding the words: "or a mixture of air and carbon dioxide" after the words "cold dry air."

The purpose of this change is to permit cooling by the direct use of carbon dioxide as is in accordance with the usual practice when dry ice cold boxes are employed.

Tentative Specifications for GR-M Polychloroprene Sheath Compound for Electrical Insulated Cords and Cables (**D 752 – 44 T**):[2]

Section 4.—Change the requirement for depreciation in tensile strength and elongation at rupture after 18 hrs. in oil immersion test at 249 to 251 F. from 35 per cent maximum depreciation to a requirement that the tensile strength and elongation after the test shall be not less than 60 per cent of their original values. This change is recommended by the committee as being more in line with actual practice than the present requirement in this severe test.

Tentative Specifications for Cellular Rubber Products (**D 798 – 46 T**).[3]—Revise Tables I, II, III, and IV of these specifications deleting all reference to the values for compression set using 25 per cent deflection as now shown under Suffix B. This change affects grades numbered 14 and 15 in each table. In place of the set values under 25 per cent deflection as now shown for these grades, add set values in the 50 per cent deflection column which shall be numerically the same as the values now given for grades numbered 13 in each table. The committee has compared compression sets on 30 samples determined at both 25 per cent and 50 per cent deflection and has found by statistical analysis of the results that a definite relationship exists for sponge rubbers having a compression set of less than 12 per cent at 50 per cent deflection. In this case, the set at 50 per cent deflection was approximately 1.38 times that at 25 per cent deflection. However, with sponge rubbers having sets greater than 12 per cent, ratios as high as 3.3 to 1.0 were found. The committee, therefore, feels that compression set values obtained at

25 per cent deflection may give misleading information with respect to sponge which might be used at deflections greater than 25 per cent and that deletion of the 25 per cent deflection requirements will be desirable.

Tentative Method of Test for Permeability of Vulcanized Rubber or Synthetic Elastomers to Volatile Liquids (**D 814 - 44 T**):[2]

Section 2 (b).—Change the specification of the inside diameter of the permeability jar from "2.328 ± 0.015 in." to read "2.375 ± 0.015 in." The dimension now given is not in conformance with that of the standard jar commercially available.

Tentative Methods of Testing Rubber Adhesives (**D 816 - 44 T**):[2]

Sections 7 (b), 10, and 18.—Change the aging period after bonding from "3 hr.," as now specified to read "24 hr." The committee has found that the 3-hr. period is not sufficiently long to allow for adequate drying and development of full bond strength in all cases.

Section 13.—Change the last sentence and add an additional sentence to read as follows: "The load so indicated expressed in pounds per inch of width for separation at 1 in. per min. shall be considered as the adhesion strength by the stripping method. For the evaluation of each cement sample, two samples shall be prepared and tested and the higher average load obtained shall be reported as the adhesion strength of the sample."

In this case, the committee is of the opinion that check tests which have not been required should be run on all samples to assure satisfactory results.

Tentative Recommended Practice for Conditioning of Rubber and Plastic Materials for Low-Temperature Testing (**D 832 - 45 T**).[3]—The committee recommends that additional Sections 10 to 17, inclusive, covering low-temperature cabinets as given in the Appendix[4] to this report be added in this tentative. A large number of types of cold boxes and low-temperature conditioning equipment are available and are being used. It is felt that the discussion of this equipment and the standardization of the essential requirements as given in this recommendation will be very helpful in improving the quality of low-temperature testing.

II. ADOPTION OF TENTATIVES AS STANDARD

The following two tentatives are recommended for adoption as standard without revision and the committee accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society:

Tentative Method of Test for Compression-Deflection Characteristics of Vulcanized Rubber (D 575 - 40 T)[2] and

Tentative Method of Test for Young's Modulus in Flexure of Natural and Synthetic Elastomers at Normal and Subnormal Temperatures (D 797 - 44 T).[2]

These two tentatives have been published for several years and are in regular use. No criticisms or suggestions for improvement have been received by the committee. The committee also recommends that the Tentative Method of Testing Compressed Asbestos Sheet Packing (D 733 - 43 T)[2] be adopted as standard with a minor revision in the type of micrometer gage specified for the measurement of thickness. The committee feels that better thickness measurements of this type of material will be obtained if both the presser foot and the anvil of the micrometer gage are $\frac{1}{4}$ in. in diameter instead of having the material rest on a larger plate anvil as at present.

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[4] These revised recommended practices and methods were accepted by the Society and appear in the 1946 Book of A.S.T.M. Standards, Part III-B.

Section 5.—Change to read as follows: "Thickness shall be determined in accordance with the procedure adopted in Section 8 (*d*) of Methods D 412, except that the thickness recorded shall be the average of not less than five separate measurements; the presser foot shall exert a force of 9.0 ± 0.1 oz.; and the diameter of both presser foot and anvil shall be 0.25 ± 0.01 in."

III. REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions in the following two standards and asks that these modifications be approved for reference to letter ballot of the Society for adoption as standard:

Standard Methods of Test for Abrasion Resistance of Rubber Compounds (**D 394 – 40**):[2]

Section 9.—Change the speed of rotation of the rubber-coated metal drum from "40 r.p.m." to read "45 ± 5 r.p.m." The manufacturer of this machine has indicated that some tolerance in speed is required and that use of standard motors necessitates this change. The effect of the changed speed has been checked by the National Bureau of Standards and found to make no material difference in the result of the test.

Standard Methods of Testing Rubber Insulated Wire and Cable (**D 470 – 41**).[2]—Replace Section 29 on test specimens for the ozone resistance test with the new Section 29 as appended hereto.[4] The present section has led to some misunderstanding and Subcommittee V on Insulated Wire and Cable has prepared the present revision in an effort to clarify the questionable points.

IV. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD

The committee recommends that the tentative revision of the Standard Specifications for Insulated Wire and Cable: Heat-Resisting Rubber Compound (D 469 – 41) now published as Table IV be adopted as standard, replacing the present Table III on insulation resistance. This table is based on a constant of 10,560 instead of 15,840. No adverse comments have been received since the publication of this tentative revision.

V. REVISION OF STANDARDS AND REVERSION TO TENTATIVES

The committee recommends that the Standard Specifications for Friction Tape for General Use for Electrical Purposes (D 69 – 38)[2] and the Standard Specifications for Rubber Insulating Tape (D 119 – 38)[2] be reverted to tentative and revised to include most of the provisions now published as Emergency Alternate Provisions EA – D 69b and EA – D 119. The emergency alternate provisions should then be withdrawn.

The new Tentative Specifications for Friction Tape will agree with the present Standard Specifications D 69 as modified by EA – D 69b except for the following: Paragraph (*a*) of Section 20 shall remain the same as in the present standard but Paragraph (*b*) of the same section should be omitted as indicated in the emergency alternate provisions. The new Tentative Specifications for Rubber Tape are to agree with the present standard Specifications D 119 as modified by EA – D 119 except for the following:

Section 4.—Change to read as follows: "The rubber compound shall contain not more than 7 lb. of new rubber per 27,000 sq. in. of tape 0.027 in. in thickness."

Section 13.—Change to read as follows:

13. *Aging and Storage.* (*a*) The tape shall at any time within four months after acceptance, if properly stored, meet the fusion test specified in Section 20 and shall not become unduly attached to the separator.

(*b*) Tape, to be properly stored, shall be stored in the original boxes and preferably in a cool, dark location. Tape shall not be stored in close proximity to steam pipes, radiators, or other sources of heat.

All of the proposed changes have been included in the revised specifications which are appended hereto.[5]

VI. REVERSION OF STANDARDS TO TENTATIVE

The committee recommends that the Standard Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (**D 27 – 41**) and Methods of Testing Rubber Insulated Wire and Cable (**D 470 – 41**) be reverted to the status of tentative as it plans to revise these standards to incorporate certain of the emergency alternate provisions issued as EA – D 27 and EA – D 470.[6]

VII. TENTATIVES CONTINUED AS TENTATIVE WITHOUT REVISION

The committee has no new tentatives to recommend for publication at the present time but is actively working on many new projects and on revisions of existing tentatives which are not mentioned in this report. The committee recommends that all tentatives under its jurisdiction, other than those referred to herein be continued as tentative without revision until the work under way can be completed.

The recommendations appearing in this report have been submitted to letter ballot of the committee, which consists of 156 voting members; 110 members returned their ballots, with the results shown in Table III.

ACTIVITIES OF SUBCOMMITTEES

Two new subcommittees were organ-

TABLE III.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. REVISION OF TENTATIVES			
Test for Compression Set of Vulcanized Rubber (D 395 – 40 T)	77	0	33
Testing Cellular Rubber Products (D 552 – 46 T)	49	1	60
Testing Asphalt Composition Battery Containers (D 639 – 43 T)	31	1	78
Test for Indentation of Rubber by Means of the Durometer (D 676 – 44 T)	77	0	33
Spec. for Rubber and Synthetic Rubber Compounds for Automotive and Aeronautical Applications (D 735 – 43 T)	51	1	58
Test for Low-Temperature Brittleness of Rubber and Rubber-Like Materials (D 736 – 43 T)	73	0	37
Spec. for GR-M Polychloroprene Sheath Compound for Electrical Insulated Cords and Cables (D 752 – 44 T)	49	0	61
Spec. for Cellular Rubber Products (D 798 – 46 T)	48	0	62
Test for Permeability of Vulcanized Rubber or Synthetic Elastomers to Volatile Liquids (D 814 – 44 T)	63	0	47
Testing Rubber Adhesives (D 816 – 44 T)	57	1	52
Rec. Practice for Conditioning of Rubber and Plastic Materials for Low-Temperature Testing (D 832 – 45 T)	75	0	35
II. ADOPTION OF TENTATIVES AS STANDARD			
Test for Compression-Deflection Characteristics of Vulcanized Rubber (D 575 – 40 T)	68	0	42
Test for Young's Modulus in Flexure of Natural and Synthetic Elastomers at Normal and Subnormal Temperatures (D 797 – 44 T)	60	0	50
Testing Compressed Asbestos Sheet Packing (D 733 – 43 T)	42	0	68
III. REVISION OF STANDARDS, IMMEDIATE ADOPTION			
Test for Abrasion Resistance of Rubber Compounds (D 394 – 40)	69	0	41
Testing Rubber Insulated Wire and Cable (D 470 – 41)	55	1	54
IV. ADOPTION AS STANDARD OF TENTATIVE REVISION OF STANDARD			
Spec. for Insulated Wire and Cable: Heat-Resisting Rubber Compound (D 469 – 41)	50	0	60
V. REVISION OF STANDARDS AND REVERSION TO TENTATIVE			
Spec. for Friction Tape for General Use of Electrical Purposes (D 69 – 38)	57	0	53
Spec. for Rubber Insulating Tape (D 119 –38)	57	0	53
VI. REVERSION OF STANDARDS TO TENTATIVE			
Spec. for Insulated Wire and Cable: Class Ao, 30 per cent Hevea Rubber Compound (D 27 – 41)	52	0	48
Testing Rubber Insulated Wire and Cable (D 470 – 41)	53	0	47

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[5] These revised specifications were accepted by the Society and appear in the 1946 Book of A.S.T.M. Standards, Part III-B.

[6] See Editorial Note, p. 423.

ized during the year. One of these, Subcommittee XXVI on Plasticity Tests, will investigate means and methods for determining the processibility of rubber and rubber-like materials. V. L. Smithers was appointed chairman and has already held an organization meeting at which time work was started on the collection of information concerning the types of instruments and methods now in use in industry and the results obtained with them in judging the processing characteristics of uncured stocks. This kind of information will provide a basis for subsequent development of standard equipment and test procedures. The other new Subcommittee XXVII on Tests of Resilience, is under the chairmanship of E. G. Kimmich and also has held its organization meeting. This subcommittee intends to develop standard definitions of terms, methods of tests, and specifications pertaining to resilience, damping, and dynamic stiffness of rubber and rubber-like materials. The subcommittee is currently engaged in developing suitable definitions of the terms used in connection with resilience tests and has created a section which is studying the three main classifications of tests frequently used, namely, rebound, free vibration, and forced vibration. The subcommittee feels that slow-speed load-deflection tests often referred to as "static" tests have little value in its work.

There have been several changes of subcommittee chairmen during the year. A. M. Finley was appointed chairman of Subcommittee I on Hose replacing E. G. Kimmich who resigned to organize the new Subcommittee on Resilience. Gordon Thompson has accepted the chairmanship of Subcommittee IV currently titled Protection of Persons from Electric Shock replacing H. S. Vassar who is retiring. R. H. Taylor has been appointed chairman of Subcommittee XIV on Abrasion Tests replacing G. S. Haslam who resigned because of his changed interests. The committee deeply appreciates the long and loyal service of the retiring chairmen and the willing cooperation of the new appointees. All of the subcommittees have been very active during the year but it is possible in this report to refer specifically to important work in progress in only a few of them.

Technical Committee A on Automotive Rubber (W. J. McCortney, chairman, E. G. Kimmich, secretary).—Technical Committee A has held four meetings since the 1945 report and in addition, many of its sections have met separately throughout the year. These meetings have been very well attended indicating strong continuing interest in the work of this committee. Although the committee limits its work to automotive applications of rubber products, it cooperates with other Committee D-11 subcommittees in promoting their work in the automotive industry. Thus, the A.S.T.M. Specifications for Cellular Rubber (D 798) developed by Subcommittee XXII have been approved by the Technical Committee and referred for publication by the Society of Automotive Engineers as well as A.S.T.M. Standards for four types of fuel and oil hose and for a number of types of coolant hose have been established for the automotive industry. The rubber classifications developed by the Technical Committee and published in the Specifications for Rubber and Synthetic Rubber Compounds (D 735) have continued to gain increasing acceptance in the automotive field. The committee is continuing to review these specifications and expects to recommend certain additional revisions in the near future so as to meet changed circumstances in the trade and to correct inadequacies. It should be remembered that these rubber classifications were developed entirely from the viewpoint of automotive use and in most cases, are not generally applicable to needs of other industries. However, these specifications have been utilized by both the Army and Navy as a basis for their specifications on automotive requirements. During the war, practically all effort of the committee has been directed toward helping the military forces with their automotive specifications and development problems. Appreciation of the work of the committee has been evidenced by the citation of the committee and its members by the Navy Department for a distinguished service award. Cooperation with government agencies is continuing although the principal effort of the committee is now directed toward peacetime uses. Additional work is in progress in the committee on V-belts, hydraulic brake cups, gaskets, oil seals, engine mounts and other vibration insulators, bumpers, and ordnance lubricating gun hose.

Subcommittee XI on Chemical Analysis of Rubber Products (S. Collier, chairman).—Further investigation is continuing on methods of analysis of synthetic elastomers since the publication of the Tentative Methods D833.[6] Methods of detection of elastomers which have been proposed have been evaluated for accuracy, speed, and ease of manipulation using individual elastomer stocks and mixtures. The more rapid spot tests are satisfactory for the detection of individual elastomers, but are useful for mixtures only in a limited number of cases. The slower pyrolysis tests are equally good for the detection of individual elastomers, and somewhat more useful than spot tests on certain mixtures. In general, natural rubber—GR-S mixtures are difficult to identify as such, since the tests display the characteristics of the major component. The spot methods have possibilities for use in sorting scrap rubber. Work is also in progress on improved methods for the determination of sulfur. It is expected that this will eventually lead to revision of the present standard methods.

Subcommittee XV on Aging Tests (H. E. Outcault, chairman).—This subcommittee has created four separate sections which are investigating aging tests currently used and possible changes which may be needed. One of these sections is studying methods of aging the rubber-like material, polyvinyl chloride, and will seek the cooperation in this of Committee D-20 on Plastics. Another section is investigating the present air oven testing methods and any changes that may be needed. The third section is studying aging data which have been accumulated on GR-S types of synthetic rubber and hopes to recommend improved methods for these materials. The fourth section is endeavoring to cover the broad field of the correlation and effect of various other miscellaneous properties of rubber and rubber-like materials upon their aging life. This work has a long range objective of covering thoroughly the problems involved in testing the aging life of many new types of synthetic materials and should lead to very constructive revisions of present methods.

Subcommittee XVII on Tests of Hardness, Set, and Creep (L. V. Cooper, chairman).—This subcommittee is continuing its investigation of the currently used methods of testing compression set with particular attention to the effect of the different variables involved. This work will undoubtedly result in some revision of the present procedures. The subcommittee is also giving attention to a new type of durometer known as the Rex hardness gage which is finding considerable use replacing the Shore durometer. The committee finds that the Rex gage is desirable because of its compactness and because it gives and holds the instantaneous hardness reading. Numerically, the readings are close to those obtained with the Shore durometer. On the other hand, it appears that the graduations of the Rex gage are too coarse, that the instrument cannot be satisfactorily calibrated by the user and that specific instructions for its use covering such points as a standard rate of application have not been developed. It is expected, however, that provision for the use of the Rex gage will be included in the present standard hardness testing methods in the near future. The subcommittee has also organized a Section on Creep Determination which has started work on methods of testing this important property.

Subcommittee XX on Adhesion Tests (A. H. Flower, chairman).—This subcommittee is starting a review of the adhesion test methods at present under its jurisdiction as well as an investigation of other adhesion methods used for the same purpose. It is also of the opinion that there could be better terminology for expressing the results in adhesion work and this subject will be given further study. In the testing of the adhesion of rubber to metal by the stripping method, the committee believes that an investigation should be made as to the effect of the position and angle of shear on the result of the test. A section has been organized to undertake this study.

Subcommittee XXI on Tests of Liquid Rubber Products (J. F. Anderson, chairman).—The subcommittee has reopened the question of viscosity test methods for cements because it has learned that present Standard Methods D 553 are now somewhat limited in use and many other methods are being employed. This whole field is again being surveyed and the most promising additional methods will be thoroughly investigated. The committee has also decided that the shear-type adhesion test which is given as method B in the Tentative Methods of Testing Rubber Adhesives (D 816) should be given further study and a special section has been appointed for this purpose. This subcommittee is now very actively engaged in the development of classification tables for specification purposes covering cements, using a somewhat similar plan to that employed in the Tentative Specifications for Rubber and Synthetic Rubber Compounds for Automotive and Aeronautical Applications (D 735). A proposed outline of such a specification has been prepared and a cooperative test program using specially prepared cements of known quality is being arranged for the purpose of establishing ranges and limits for the various specified values.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, Simon Collier.
Vice-Chairman, H. E. Outcault.
Secretary, Arthur W. Carpenter.
Members-at-Large of Advisory Subcommittee:
L. V. Cooper,
O. M. Hayden,
R. A. Schatzel, and
T. A. Werkenthin.

This report has been submitted to letter ballot of the committee, which consists of 156 members; 110 members returned their ballots, of whom 96 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

SIMON COLLIER,
Chairman.

ARTHUR W. CARPENTER,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-11 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

- Rubber Sheath Compound for Electrical Insulated Cords and Cables (D 532 - 46 T),
- Insulated Wire and Cable: Ozone-Resistant Type Insulation (D 574 - 46 T),
- Insulated Wire and Cable: Heat-Resistant Synthetic Rubber Compound (D 754 - 46 T),
- Insulated Wire and Cable: Performance Synthetic Rubber Compound (D 755 - 46 T), and
- Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (D 27 - 46 T).

Revision of Tentative Methods of:

- Testing Rubber Insulated Wire and Cable (D 470 - 46 T),
- Testing Rubber Hose (D 380 - 46 T),
- Testing Rubber-Coated Fabrics (D 751 - 46 T), and
- Identification and Quantitative Analysis of Synthetic Elastomers (D 833 - 46 T).

These recommendations were accepted by the Standards Committee on September 9, 1946, and the revised tentatives appear in the 1946 Book of A.S.T.M. Standards, Part III-B, bearing the designations indicated above.

The emergency alternate provisions referred to earlier in the report were discontinued by action of the Standards Committee on September 9, 1946.

REPORT OF COMMITTEE D-12*

ON

SOAPS AND OTHER DETERGENTS

No meetings of Committee D-12 on Soaps and Other Detergents were held during 1945 and the first meeting since 1944 was held in New York, N. Y., on March 18 and 19, 1946.

There have been a number of changes in membership and representatives. The committee now consists of 92 members, of whom 42 are classified as producers, 25 as consumers, and 25 as general interest members.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, B. S. Van Zile.
Vice-Chairman, F. W. Smither.
Secretary, J. C. Harris.
Advisory Committee:
B. S. Van Zile
W. G. Morse
W. H. Koch
C. C. Zeigler
J. B. Crowe
F. Krassner
M. L. Sheely
J. C. Harris
F. W. Smither

RECOMMENDATIONS AFFECTING STANDARDS

The committee is recommending revisions in one tentative, seven tentative revisions of standards, adoption of one tentative as standard, adoption of two tentative revisions of standards, withdrawal of nine emergency alternate provisions and reaffirmation of four existing standards. The standards affected are listed in Table I and the recommendations are explained in the reports of the subcommittees directly responsible for them.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Methods of Testing (J. B. Crowe, chairman).—Section A on Methods of Soap Analysis (E. Randa, acting chairman) recommended that the Tentative Methods for the Analysis of Soap Containing Synthetic Detergents (D 820 – 45 T)[1] be adopted as standard with the following editorial changes:

Section 8(d).—In the last sentence, after the word "cool" add the phrase "in a desiccator."

Section 18(d).—Change the first sentence to read as follows: "Evaporate on a steam bath until the odor of petroleum ether is no longer perceptible and then dry for ½ hr. in an oven at 105 ± 2 C."

The subcommittee recommended that the tentative revision[2] of the Standard Methods of Sampling and Chemical Analysis of Soaps and Soap Products (D 460 – 44) be adopted as standard. This revision covers the addition of a procedure for determining free alkali and potassium carbonate in potash paste soaps.

Section A recommended the following editorial correction in the Standard Methods of Sampling and Chemical Analysis of Soaps and Soap Products (D 460 – 44):

Section 32(a).—Change the formula for the calculation of the determination of rosin in soap from its present form: namely,

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

[1] 1945 Supplement to Book of A.S.T.M. Standards, Part III.

[2] 1944 Book of A.S.T.M. Standards, Part III.

$$R_S = \frac{R_1 \times 1.064 \times A}{100}$$

to read:

$$R_s = R_2 \times 1.064$$

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. Revision of Tentative			
Def. of Terms Relating to Soaps and Other Detergents (D 459 – 45 T)	54	0	12
II. Tenative Revisions of Standards			
Spec. for Milled Soap (D 455 – 39)	53	0	13
Spec. for Chip Soap (D 496 – 39)	54	0	12
Spec. for Powdered Soap (D 498 – 39)	54	0	12
Spec. for White Floating Toilet Soap (D 499 – 39)	53	0	13
Spec. for Olive Oil Solid Soap (D 592 – 42)	53	0	13
Spec. for Olive Oil Chip Soap (D 630 – 42)	53	0	13
Spec. for Chip Soap with Rosin (D 690 – 44)	53	0	13
III. Adoption of Tentative as Standard			
Chemical Analysis of Soaps Containing Synthetic Detergents (D 820 – 45 T), as revised	56	0	10
IV. Adoption as Standard of Tentative Revisions of Standards			
Chemical Analysis of Special Detergents (D 501 – 44)	56	0	10
Chemical Analysis of Soap (D 460 – 44)	55	0	11
V. Reaffirmation of Existing Standards			
Spec. for Caustic Soda (D 456 – 39)	57	0	9
Spec. for Modified Soda (D 457 – 39)	56	0	10
Spec. for Soda Ash (D 458 – 39)	57	0	9
Spec. for Ordinary Bar Soap (D 497 – 39)	53	1	3

Section 59(b).—Change the starch factor from "0.90" to read "0.93."

Section C of Subcommittee I and Section D of Subcommittee II on Dry Cleaning Detergents (G. P. Fulton, chairman) reported that an effort will be made to enlist the cooperation of manufacturers of dry-cleaning soaps and it is intended to draw up a classification of various types of dry-cleaning soaps to arouse interest in the work of the section to aid in the preparation of tentative specifications.

Section D on Special Detergents (W. H. Koch, chairman) reported that the preparation of analytical methods will be initiated for sodium bicarbonate and borax.

The section recommended that the tentative revision[2] of the Standard Methods of Sampling and Chemical Analysis of Special Detergents (D 501 – 43) be adopted as standard.

Section E on Physical Testing (J. A. Woodhead, chairman) reported considerable interest in the development of a standard method for determining detergency, but that no unanimity of opinion was developed. A survey of the literature will be made with the object of reducing the numerous methods to a series of factors or to a few methods more amenable to control. This survey can serve as the ground work upon which it is hoped a standard detergency test may be developed.

Subcommittee II on Specifications (F. W. Smither, chairman).—Section A on Soap Specifications (Frederick Krassner, chairman) is now the consolidated committee which heretofore comprised Sections on Straight, Built and Textile Soaps. Consideration was given to the disposition of the emergency alternate provisions which, even though declared eliminated after July 1, 1946, by the Executive Committee of the Society, are yet required in view of the demands of the present situation. The specifications involved are those for Milled Toilet Soap (D 455 – 39), Chip Soap (D 496 – 39), Powdered Soap (D 498 – 39), White Floating Toilet Soap (D 499 – 39), Olive Oil Solid Soap (D 592 – 42), Salt-Water Soap (D 593 – 42), and Olive Oil Chip Soap (D 630 – 42). It was recommended that these emergency alternate provisions[2] be published as tentative revisions of the standards. Further recommendations were that the matter insoluble in water be changed for white floating soap (D 499 – 39) from "0.2 per cent" to "0.4 per cent" and for milled toilet soap (D 455 – 39) from "0.4 per cent" to "0.6 per cent."

The subcommittee recommended that the following tentative revision in the Standard Specifications for Compound Chip Soap (With Rosin) (D 690 – 44)[2] be accepted:

Section 3.—Change the percentage of matter volatile at 105 C. from "12 per cent" to read "15 per cent."

Section F on Special Detergents (C. C. Ziegler, chairman) recommended that since the following specifications still represented general practice and required no revision they should be reapproved:

Specifications for:

Soda, Caustic (D 456 – 39),
Soda, Modified (Sesquicarbonate Type) D 457 – 39),
Soda Ash (D 458 – 39), and
Ordinary Bar Soap (D 497 – 39).

Section G on Metal Cleaners (J. C. Harris, chairman) reported that the suggested immersion corrosion test for aluminum cleaners is to be revised. A test will be developed to demonstrate the corrosive effect of residual cleaner. It was proposed that the immersion corrosion test be subjected to cooperative evaluation.

Subcommittee III on Nomenclature and Definitions (C. A. Marlies, chairman) recommended that the Tentative Definitions of Terms Relating to Soaps and Other Detergents (D 459 – 45 T)[1] be revised as follows:

Soap.—In Note (*c*) delete the word "physical."

Penetrating Agent, Surface Active Agent, and *Wetting Agent.*—In each of these three definitions change "composition" to read "material."

Change the following definitions to read:

Dry Cleaning.—Cleaning fabrics in a substantially nonaqueous liquid medium.

Washing.—Cleaning in a liquid medium.

Wet Cleaning.—A term used in the dry cleaning industry to denote cleaning in an aqueous medium.

New Definitions.—Add the following as new definitions:

Buffer Action.—The resistance of a solution to change in pH.

Dispersing Agent.—A material which increases the stability of a suspension of particles in a liquid medium.

Emulsifying Agent.—A material which increases the stability of a dispersion of one liquid in another.

Subcommittee IV on Papers and Publications (W. H. Koch, chairman) decided after much discussion that the paper by W. G. Morse on "Cleaning and Cleaning Materials" because of its controversial nature should perhaps be published under the auspices of the National Association of Purchasing Agents.

A paper by J. C. Harris on "The Evaluation of Surface Active Agents"[3] was to be revised and submitted to the Society for publication.

The special compilation "A.S.T.M. Standards on Soaps and Other Detergents," was reissued in September, 1945, in which, in addition to the usual standards and tentatives, there are included tentative methods for the chemical analysis of soap containing synthetic detergents, chemical analysis of special detergents, chemical analysis of sulfonated oils, and the analysis of industrial metal cleaning compositions. As additional information there is included an Annotated Bibliography of Aluminum Cleaning brought up to date by the authors. J. C. Harris and R. B. Mears.

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[3] ASTM BULLETIN, Am. Soc. Testing Mats., No. 140, May, 1946, p. 76.

This report has been submitted to letter ballot of the committee, which consists of 92 members; 57 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

B. S. VAN ZILE,
Chairman.

J. C. HARRIS,
Secretary.

EDITORIAL NOTE

The emergency alternate provisions mentioned earlier in the report were discontinued by action of the Standards Committee on September 9, 1946.

REPORT OF COMMITTEE D-13* ON TEXTILE MATERIALS

Committee D-13 on Textile Materials held one meeting during the year in New York, N. Y., on March 13 to 15, 1946. The usual fall meeting was canceled on account of the difficulty of securing adequate hotel accommodations. The registered attendance of 222 members and guests indicated that interest in the work of the committee had not waned even after a lapse of seventeen months since the last meeting. The Advisory Committee and twenty of the subgroups of Committee D-13 held meetings to consider work which had been carried on by correspondence, and to lay plans for future activities.

The following changes have been made in the organization of Committee D-13: in Subcommittee A-1, Section V on Heavy Cotton Woven Fabrics has been discontinued and its field combined with that of Section III, the title of which is changed to Section III on Cotton Woven Fabrics; in Subcommittee A-8, Section IV on Chemical Tests has been discontinued, but a Section V on Abrasion and a Section VI on Sizing have been added; Subcommittee C-2 on Papers and Publicity has been divided into two parts to be known as Subcommittee C-2 on Papers and Subcommittee C-4 on Publicity; a new Subcommittee C-5 on Significance of Tests has been authorized.

Dean Harvey, who has retired from active business, has been elected an honorary member of Committee D-13 in recognition of his long, faithful, and efficient service to the committee.

L. S. Converse, a member of Committee D-13, is serving as a consultant to the Society's new Committee E-9 on Fatigue.

Originating from an inquiry by the Technical Committee of the Institute of Carpet Manufacturers for methods of test of kraft paper yarns, Committee D-6 on Paper and Paper Products has indicated its feeling that Committee D-13 should undertake the development of such methods. This project has been assigned to Subcommittee A-3, Section IV on Pile Floor Coverings, since such yarns find limited use by the manufacturers of floor coverings.

A study has been initiated looking to the general improvement of the standards sponsored by Committee D-13. Attention will be focused upon securing a more carefully defined content of "scope" clauses, and upon recognizing and setting up the general content of a standard in a more logical manner. This study has been assigned to Subcommittee B-5 on Sampling, Presentation and Interpretation of Data.

RECOMMENDATIONS AFFECTING STANDARDS

Committee D-13 is presenting for publication as tentative two new methods of test and is recommending the revision of five tentatives, revision of two standards and reversion to tentative, tentative revisions of four standards, revision for immediate adoption of one standard, adoption as standard of three tentatives, withdrawal of two emergency alternate provisions, and the reapproval of eight standards which have stood six years without revision or reaffirmation.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

The revisions being presented are given in detail in the Appendix and are referred to, where necessary, under the subcommittee activities. The new tentatives and existing standards affected are listed in Table I.

These recommendations have been submitted to letter ballot of the committee, which consists of 306 members, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. New Tentatives			
Tests and Tolerances for Rayon Tire Cord	58	5	115
Test for Compatability of Glass Yarn with Insulating Varnish	32	0	146
II. Revisions of Tentatives			
Spec. for Woven Asbestos Cloth (D 677 – 42 T)	38	0	140
Testing and Tolerances for Glass Yarn (D 578 – 44 T)	44	0	134
Quantitative Analysis of Textiles (D 629 – 42 T)	134	0	44
Def. of Terms Relating to Textile Materials (D 123 – 45 T)	142	4	32
Rec. Practice for a Universal System of Yarn Numbering (D 861 – 45 T)	119	6	53
III. Revision of Standard and Reversion to Tentative			
Testing and Tolerances for Tire Cord, Woven and on Cones (D 179 – 42)	57	3	118
Testing and Tolerances for Continuous Filament Rayon Yarns (D 258 – 44)	75	1	102
IV. Tentative Revisions of Standards			
Test for Fineness of Wool (D 419 – 44)	61	0	117
Spec. and Test for Fineness of Wool Tops (D 472 – 41)	58	0	120
Test for Hard Scoured Wool in Wool in the Grease (Laboratory Scale Operations) (D 584 – 43)	55	0	123
Spec. for Textile Testing Machines (D 76 – 45)	116	1	61
V. Adoption of Tentatives as Standard			
Testing and Tolerances for Rope (Leaf and Bast Fibers) (D 738 – 45 T)	39	1	138
Testing and Tolerances for Spun, Twisted, or Braided Products Made from Flax, Hemp, Ramie, or Mixtures Thereof (D 739 – 45 T)	44	0	134
Test for Air Permeability of Textile Fabrics (D 737 – 43 T)	80	0	98
VI. Revisions of Standards, Immediate Adoption			
Testing and Tolerances for Knit Goods (D 231 – 39)	57	0	121
VII. Reaffirmation of Standards			
Spec. and Test for Osnaburg Cement Sacks (D 205 – 39)	29	1	148
Spec. and Test for Cotton Goods for Rubber and Pyroxylin Coating (D 334 – 40)	55	0	123
Testing and Tolerances for Certain Carded Cotton Gray Goods (D 433 – 39)	62	0	116
Spec. for 0.007-in. Cotton Tape for Electrical Purposes (D 335 – 36)	30	0	148
Test for Strength of Rayon Woven Fabric when Wet (D 415 – 38)	74	0	104
Test for Maximum Residual Shrinkage of Silk and Rayon Woven Fabrics (D 416 – 39)	63	4	111
Test for Fiber Length of Wool (D 519 – 40)	59	0	119
Spec. for Terry (Turkish) Toweling (D 505 – 40)	43	1	134

Activities of Subcommittees

Many important projects are under consideration and are mentioned briefly in the following summary of subcommittee activities.

Subcommittee A-1 on Cotton and Its Products (K. B. Cook, chairman):

Section I on Cotton (M. E. Campbell, chairman) is undertaking an exhaustive study of the Tentative General Methods of Testing Cotton Fibers (D 414 – 40 T) with a view to revision of present methods where needed and the addition of new methods. The section is recommending, as an editorial change, that the requirement for capacity of the tension testing machine be changed from "150 lb." to "300 lb." in Section 24 (*a*), on Tensile Strength.

Section II on Cotton Yarns and Threads (W. R. Marsden, chairman) has under way a thorough revision of Standard General Methods of Testing and Tolerances for Cotton Yarns (D 180 – 44).

Section III on Cotton Woven Fabrics (R. T. Fisher, chairman) now has jurisdiction over both light- and heavy-woven cotton fabrics. The following standards having been reviewed and found to be in accord with present practice are recommended for reapproval:

Spec. and Methods of Test for Osnaburg Cement Sacks (D 205 – 39),

Spec. and Test for Cotton Goods for Rubber and Pyroxylin Coating (D 334 – 40), and

Testing and Tolerances for Certain Carded Cotton Gray Goods (D 433 – 39).

Section IV on Tire Fabrics (F. C. Kennedy, chairman) recommends for publication the proposed Tentative Methods of Testing and Tolerances for Rayon Tire Cord;[1] also, a complete revision of the Standard Methods of Testing and Tolerances for Tire Cord, Woven and on Cones (D 179 – 42) and its reversion to tentative. The revised method is appended hereto.[2]

The section is giving consideration to a microscopic method for gage determination and to certain methods designed for commercial acceptance tests.

Section V on Heavy Cotton Woven Fabrics has been discontinued and its activities combined with Section III.

Section VI on Narrow Fabrics (F. S. Mapes, chairman) recommends the reapproval of Standard Specifications D 335 as being in accord with present practice.

The section is studying the standardization of woven cotton tapes and tubular sleevings and braids, the need for test methods or standards for fastner tapes, and the inclusion of tapes made from all fibers, (except asbestos and glass) in Standard Methods of Testing and Tolerances for Woven Tapes (D 259 – 44).

Subcommittee A-2 on Rayon and Its Products (A. M. Tenney, chairman):

Section I on Rayon (J. B. Goldberg, chairman) recommends the revision and reversion to tentative of the Standard Methods of Testing and Tolerances for Continuous Filament Rayon Yarns (D 258 – 44) as appended hereto.[2]

Section II on Rayon Fabrics (G. H. Hotte, chairman) recommends the reapproval of Standard Methods D 415 and D 416 as being in accord with present practice.

Subcommittee A-3 on Wool and Its Products (A. G. Ashcroft, chairman):

Section I on Wool (Werner von Bergen, chairman) has recommended tentative revisions in the Standard Methods D 419, D 472, and D 584 as given in Appendix I.

The section recommends the reapproval of the Standard Method of Test for Fiber Length of Wool (D 519 – 40) as being in accord with present practice.

Studies are being conducted on improved methods of measuring fineness and on problems related to shrinkage.

Section II on Felt (G. H. Harnden, chairman) recommends that the present tentative revisions of the Standard Methods of Testing Wool Felt (D 461 – 45) be continued as tentative pending further study.

This section is working on the following projects: machines and methods for measurement of hardness, compressibility, and compressional resilience of pressed felts, tests for flame resistance, nomenclature and specifications for roll and sheet felts, and methods of analysis for synthetic fibers in felts.

Section III on Woolen and Worsted Yarns (Fred Noechel, chairman) has presented an editorial change, as given in Appendix I, in Standard Methods D 403 and D 404, calling attention to proposed trade practice rules regarding moisture regain for handknitting yarns.

The section is studying the requirement for the number of specimens to be taken for the various tests.

Section IV on Pile Floor Coverings (G. Y. Jean, chairman) is studying the following projects: methods for evaluating intangible factors of luxury, wear testing, development of test methods for kraft cord, and method of measurement to tuft length.

Section V on Woolen and Worsted Fabrics (J. N. Dalton, chairman) is working on revisions of Standard Methods of Testing and Tolerances for Certain Wool and Part Wool Fabrics (D 462 - 44) which would specify the number of specimens for strength tests and add a method for determining shrinkage in laundering. Standard D 462 is being editorially revised to limit the shrinkage in sponging test to London shrunk fabrics.

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[1] These methods were accepted as tentative by the Society and appear in the 1946 Book of A.S.T.M. Standards, Part III-A.

[2] These revised methods were accepted by the Society and appear in the 1946 Book of A.S.T.M. Standards, Part III-A.

Subcommittee A-4 on Asbestos and Its Textile Products (F. S. Mapes, chairman):

This subcommittee recommends that Tentative Specifications D 677 be revised as given in Appendix I, and that the Emergency Alternate Provision EA - D 375 which suspended requirements for magnetic rating in asbestos roving be withdrawn.

Projects before the subcommittee are: determination of theoretical weight of a cloth from cloth count and yarn weight, method of test or check for magnetite, and determination of asbestos content in high quality mixes.

Subcommittee A-5 on Bast and Leaf Fibers and Their Products (S. J. Hayes, chairman):

This subcommittee has recommended for adoption as standard the Tentative Methods D 738 and D 739.

The subcommittee also recommends that the Tentative Methods of Testing and Tolerances for Jute Rove and Plied Yarn for Electrical and Packing Purposes (D 681 - 42 T) be continued as tentative pending completion of a study on extraction methods.

Subcommittee A-6 on Garment and Household Fabrics (Ruth O'Brien, chairman):

This subcommittee has been inactive during the war period and will now renew its study of the various standards under its jurisdiction. It is, therefore, recommended that the Tentative Specifications for Bleached Wide Cotton Sheeting (D 503 - 40 T), for Bleached Cotton Broadcloth (D 504 - 41 T), for All-Wool, All-Cotton, and Wool and Cotton Blanketing (Household) (D 576 - 40 T), for Medium-Weight Cotton Corduroy Fabrics (D 625 - 41 T), and for Finished, All-Cotton, Upholstery Tapestries (D 678 - 42 T) be continued as tentative pending their study by a special committee.

The section recommends the reapproval of the Standard Specifications for Terry (Turkish) Toweling (D 505 - 40) as being in accord with present practice.

Subcommittee A-8 on Glass Fiber and Its Products (F. S. Mapes, chairman):

This subcommittee is recommending the new Tentative Method of Test for Compatibility of Glass Yarn with Insulating Varnish.[1]

The subcommittee also recommends that Tentative Methods D 578 be revised as given in Appendix I.

Projects before the subcommittee are: twist test procedure, effect of tension in winding on weight of glass yarn, effect of jaws on breaking strength of glass yarn, determination of sizing properties, statistical sampling methods, suitability of present test methods for staple fiber products, and preparation and handling of specimens for breaking strength.

Subcommittee B-1 on Methods and Machines (W. D. Appel, chairman):

Section I on Methods (W. D. Appel, chairman) recommends the adoption as standard of Tentative Methods D 737.

The section is also recommending the withdrawal of EA - D 231, and for immediate adoption, a revision, as given in the Appendix, of the Standard Methods of Testing and Tolerances for Knitted Goods (D 231 - 39) which eliminates the requirement for a specific type of extraction apparatus.

The section recommends that the Tentative Methods of Quantitative Analysis of Textiles (D 629 - 42 T) be revised as given in Appendix II.

The following standards have been reviewed and since revisions are pending, it is recommended that they be retained in their present status: Standard General Methods of Testing Woven Textile Fabrics (D 39 – 39), Tentative Methods for Identification of Fibers in Textiles (D 276 – 43 T), and Tentative Method of Test for Colorfastness to Atmospheric Gases of Dyed Cellulose Acetate Rayon (D 682 – 42 T).

The section has under consideration the following projects: hand and draping qualities of soft woven fabrics, yarn numbers in fabrics, accelerated aging, wear testing machines and procedures, soiling properties, basic fiber properties, clamps for strength testing, effective gage length in elongation tests, and test methods for elastic fibers.

Section II on Machines (H. J. Ball, chairman) recommends a tentative revision of the Standard Specifications for Textile Testing Machines (D 76 – 45), adding a procedure for verification of recorded elongation and to permit jaw gripping surfaces greater than 1 in. parallel to the direction of load application, as given in Appendix I.

This section is continuing the study of jaws and clamps.

Section III on Atmospheric Conditions (R. H. Brown, chairman) is studying drying ovens with a view to drafting drying oven specifications.

Subcommittee B-2 on Nomenclature and Definitions (A. G. Scroggie, chairman):

This subcommittee is recommending a revision of the Tentative Definitions of Terms Relating to Textile Materials (D 123 – 45 T) which withdraws the definition of protan and adds several new definitions in Part A and additional fibers in Part B as appended hereto.[2]

The subcommittee also recommends that the Tentative Recommended Practice for a Universal System of Yarn Numbering (D 861 – 45 T) be editorially revised as appended hereto.[2]

Subcommittee B-4 on Bleaching, Dyeing, and Finishing.—The work of this subcommittee has been slowed up through the resignation of J. Robert Bonnar, acting chariman. It is recommended that, pending renewal of activity, the following tentatives be retained in their present status:

Tentative Specifications for:

- Fire-Resistant Properties of Treated Textile Fabrics (D 626 – 41 T),

Tentative Methods of Test for:

- Volumetric Determination of Small Amounts of Copper in Textiles (D 463 – 37 T),
- Apparent Fluidity of Dispersions of Cellulose Fibers in Cuprammonium Hydroxide (D 539 – 40 T),
- Resistance of Textile Fibers to Water (D 583 – 40 T),
- Evaluating Compounds Designed to Increase Resistance of Fabrics and Yarns to Insect Pests (D 627 – 41 T), and
- Identification of Finishes on Textiles (D 683 – 42 T).

Subcommittee B-5 on Sampling, Presentation, and Interpretation of Data (O. P. Beckwith, chairman):

This subcommittee is actively engaged in assisting other subcommittees in developing statistically the number of tests required in various methods for desired degrees of precision. A study of Committee D-13 standards with a view to their improvement has been inaugurated.

The following officers were elected at the March, 1946, meeting for the two-year period from June, 1946, to June, 1948:

Chairman, H. J. Ball.
First Vice-Chairman, A. G. Scroggie.
Second Vice-Chairman, Martin Castricum.
Secretary, W. H. Whitcomb.

This report has been submitted to letter ballot of the committee, which consists of 306 members; 178 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. J. BALL,
Chairman.

W. H. WHITCOMB,
Secretary.

EDITORIAL NOTE

The emergency alternate provisions, EA-D 231 and EA-D 375, mentioned earlier in the report, were discontinued by action of the Standards Committee on September 9, 1946.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS ON TEXTILE MATERIALS

In this Appendix are given proposed revisions in certain standards and tentatives covering textile materials, which are referred to earlier in this report.[1] These standards and tentatives appear in their present form in the 1944 Book of A.S.T.M. Standards, Part III, and the 1945 Supplement, Part III.

REVISIONS OF TENTATIVES

Tentative Specifications for Woven Asbestos Cloth (D 677 – 42 T):

Table I.—Change to read as shown in the accompanying Table I.

TABLE I.—PHYSICAL PROPERTIES AND AVAILABLE GRADES OF ASBESTOS CLOTH.

Weight, lb. per sq. yd.	Thickness,[a] in.	Construction, yarns per in.		Yarn Number		Available Grades				
		Warp	Filling	Warp	Filling	Commercial	Underwriter's	A	AA	AAA
1.00	0.030	16	13	18/2	18/1		×			
1.05	0.030	21	15	28/2	28/2		×			
1.25	0.035	28	15	28/2	28/2		×			
1.35	0.040	16	14	18/2	18/2		×			
1.50	0.050	20	14	18/2	18/2		×			
1.65	0.055	14	14	14/2	14/2	×	×	×	×	×
1.75	0.060	26	13	18/2	18/2	×	×			
1.80	0.063	20	10	14/2	14/2	×	×	×	×	×
2.15	0.065	20	10	12/2	12/2	×	×	×	×	×
2.25	0.070	18	8	10/2	10/2	×	×	×	×	×
2.50	0.075	20	9	10/2	10/2	×	×	×	×	×
3.10	0.100	18	9	10/3	10/2	×	×	×	×	×

[a] The thickness as indicated applies to Commercial and Underwriter's grades only. It does not apply to grades with higher asbestos content, due to manufacturing variables not introduced in Commercial and Underwriter's grades.

Tentative Methods of Testing and Tolerances for Glass Yarn (D 578 – 44 T):

Section 10.—Change Paragraph (*c*) to read as follows, and delete Paragraph (*d*):

(*c*) *Number of Tests, Single and Plied Yarns.*—Each lot shall be tested by taking three packages from one of each five containers. Three twist tests shall be made on each package. The average of the nine tests shall represent the number of turns per inch of the yarn in that container. If any container fails to meet a specified twist or number of turns per inch, all containers of that group of five shall be tested.

Tentative Methods of Quantitative Analysis of Textiles (D 629 – 42 T):

It is recommended that the procedure for microscopic analysis described in Section 15 be extensively revised as shown in Appendix II.

TENTATIVE REVISIONS OF STANDARDS

Standard Methods of Test for Fineness of Wool (D 419 – 44):

Section 5.—Add the subtitle "(*a*) Long-Fiber Method.—" at the beginning of the present section. Add new Paragraphs (*b*) to (*d*) to read as follows:

(*b*) The measurement of the fibers shall be made by two operators, each measuring one half of the number of fibers required for the test, and the result for each set determined. The average of the two results shall be the value for the determination, provided that the difference between this average and each operator's result is not greater than 3 per cent of the average value. If the difference is greater than 3 per cent, the test shall be repeated and the average of all the tests shall be taken as the value for the determination.

NOTE: *Example.*—The results obtained by two operators each measuring 300 fibers are: 21.5 μ and 22.9 μ. These results differ from the average value, 22.2, by 0.7 μ, which is more than 0.666 μ or 3 per cent of the average value. A second test is necessary and each operator measures 300 additional fibers. The results obtained are: 21.9 μ and 22.3 μ. The average of all four tests, 22.2 μ, is accepted as the value for the determination.

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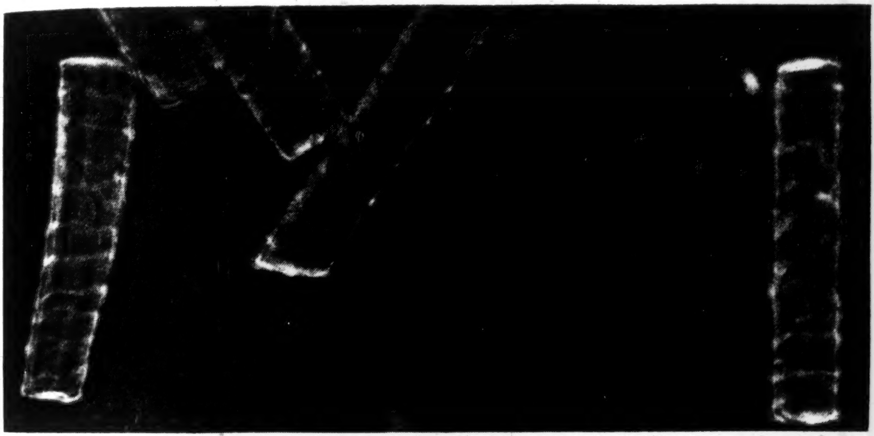
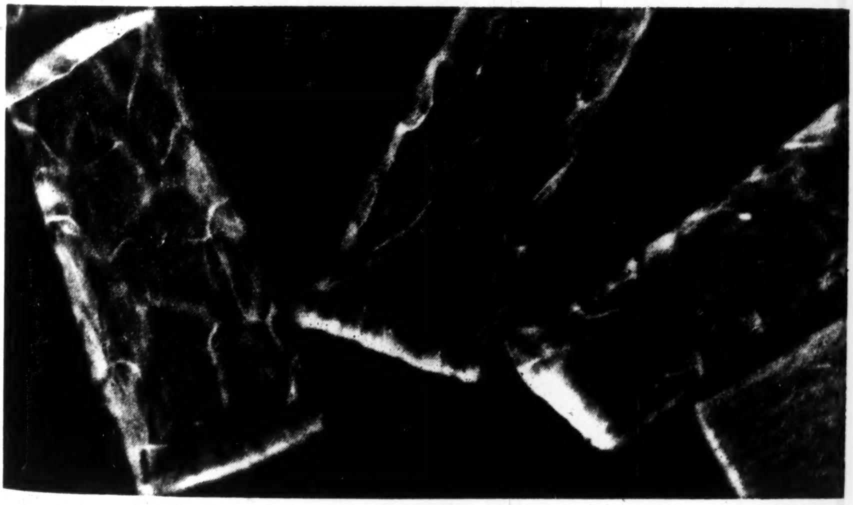
[1] See p. 428.

(c) For a comparison of two lots, the procedure described in Paragraph (b) shall be followed for each lot. Real differences in quality between the lots may be considered to exist when the lot values differ from each other by more than 3 per cent of the average and the lot values fall within different grade ranges.

NOTE: *Example.*—The results obtained from two lots are: 23.0 μ and 22.3 μ. These values differ by 0.7 μ which is more than 0.681 μ or 3 per cent of the average value, 22.7 μ. Real differences in quality of these two lots may be considered to exist since the values differ by more than 3 per cent of the average, and the values fall within different grade ranges.



(*a*) Arrangement of Fine Fibers on Slide (500×).



(*b*) Arrangement of Coarse Fibers on Slide (500×).

FIG. 1.—Dispersion of Fibers, Short-Fiber Method.

(*d*) ***Short-Fiber Method.***—The test specimen may be prepared by the following short-section method: By means of a cross-sectioning device capable of holding a sample having the approximate thickness of a top sliver or card sliver, a section of about 75 μ in length shall be made and embedded in a film of collodion or similar material. This pane of fibers shall be placed on a slide and touched with one or two drops of butyl acetate, and then, using a dissecting needle, the film shall be broken up and the fibers spread by stirring. As the butyl acetate evaporates, the slide shall be tapped occasionally to facilitate dispersion of the fibers. The adherent film, when dry, shall be covered with 2 or 3 drops of mineral oil and a cover glass. The fibers should be dispersed on the finished slide in the general manner shown in Fig. 4 (the accompanying Fig. 1). The specimen shall be measured in accordance with Paragraphs *(a)* to *(c)*, except that the fiber shall always be measured at the mid-point.

Standard Specifications and Methods of Test for Fineness of Wool Tops (D 472 – 41):

Section 9.—Change the title of this section to read "Test Specimen Preparation."

Section 10.—Make the same additions to this section as recommended above for inclusion in Section 5 of Standard Methods D 419 – 44.

Standard Method of Test for Hard Scoured Wool in Wool in the Grease (Laboratory Scale Operations) (D 584 – 43):

Section 9.—Change to read as follows:

9. A 3 to 5-g. portion, accurately weighed, of oven-dry scoured wool from each test specimen and from the tags shall be taken. Each portion shall be ignited to constant weight in a tall-form porcelain or vitreous silica crucible having the following dimensions: height, 60 mm.; top diameter, 50 mm.; bottom diameter, 25 mm.

NOTE.—Wool is likely to swell and froth when heated. To avoid loss, the sample may be burned in small portions, or it may be moistened with saturated solution of ammonium nitrate in alcohol (67 per cent), or it may be heated simultaneously at the top and bottom by a pair of gas burners.

Standard Specifications for Textile Testing Machines (D 76 – 45):

Section 2.—Add the following sentence to Note 1 under this section: "Electronic controls have been devised by means of which a constant-rate-of-traverse machine can also be operated on the constant-rate-of-load principle."

New Section.—Add new Section 6 on verification of recorded elongation, to read as follows:

6. *Verification of Recorded Elongation.*—This section provides a procedure for verification of recorded elongation which is applicable to tension testing machines of either the constant-rate-of-traverse or constant-rate-of-load class. Possible causes of error in recording elongation are given in Section 7.

(a) The clamps of the testing machine shall be returned to their normal positions for the start of a test. A chart shall be placed in the holder and its position adjusted so that the pen indicates zero load and zero elongation (Note 1). The distance between the butts of the clamps (or some other bench marks) shall be measured to the nearest 0.01 in. (Notes 2 and 3). This distance shall be designated as C_n (normal clamp distance).

NOTE 1.—On machines having a specific location for the chart as determined by supports, the chart shall be placed in its proper location and the pen adjusted to record zero load and zero elongation.

NOTE 2.—The distance can be measured conveniently by means of a pair of calipers and a steel rule graduated to 0.01 in. A cathetometer of equal precision may be used if desired.

NOTE 3.—Attention is called to the fact that the length of a specimen actually stretched is usually somewhat greater than the original specimen length measured nip-to-nip of the jaws. Elongation calculated on initial length accordingly may be somewhat in error depending on the length of specimen which is actually stretched, that is, effective gage length.

(b) The machine shall be operated without a specimen in order to register increasing elongation at zero load. If the chart is properly located in the holder, the pen line obtained in this test will be superimposed on the axis of the chart showing zero load and variable elongation. If this condition is not realized, the chart shall be adjusted until it is properly located.

NOTE 4.—If the pen is set slightly to one side of the axis line, any deviations from the parallel condition can be detected somewhat more readily than when the pen line is superimposed on the ruled axis line.

(c) With the chart properly adjusted and without a specimen in the clamps, the machine shall be operated until the pen indicates an elongation of 1.0 in. (Note 5). The distance between

the bench marks on the clamps shall be determined to the nearest 0.01 in. This distance should be equal to C_n plus the elongation recorded on the chart.

NOTE 5.—For incline plane testing machines, the plane should be left in the starting position and the carriage traversed by hand. After reaching the desired elongation, the carriage must be blocked to prevent any change in the position of the pen.

(d) This operation shall be repeated with elongations corresponding to 2.0 and 3.0 in. (Note 6).

NOTE 6.—It is not essential to use elongations of exactly 1.0, 2.0, and 3.0 in. as long as the proper increment is obtained. Thus, an indicated elongation of 1.4 in. on the chart should accompany a separation of the clamps by 1.4 in.

(e) Any discrepancy between the recorded elongation and the actual increase in distance between the clamps is an error in the recorded elongation caused by faulty spacing of the chart elongation graduations.

(f) To establish the presence of other errors in recorded elongation, a chart shall be placed in the testing machine and adjusted according to the directions given in Paragraphs (a) and (b). The clamps shall be returned to their initial positions and a load applied without permitting any separation of the clamps by gripping a nonextensible member, such as a strip of thin steel, in both clamps. The machine shall be operated so that the pen draws a line on the chart. This line should be parallel with the axis indicating variable load and zero elongation (Note 7). Failure of the pen line to parallel the axis of the chart indicates a variable error in elongation due to improper geometry of the testing machine or to the use of the incorrect angle in printing the charts.

NOTE 7.—The axis indicating zero elongation and increasing load is not necessarily a straight line. On pendulum-type testing machines, it is frequently somewhat curved at higher loads. On many testing machines, it forms an angle with the other base line indicating zero load and variable elongation. The angle depends upon the geometry of the machine.

Appendix.—Add the following as an appendix to these methods:

APPENDIX

Causes of Error in Recording Elongation

Failure of the tension testing machine to record the proper elongation may be due to one or more of the causes given in the following Paragraphs (a) to (e). One or more of these possibilities may not apply to a particular type of machine.

(a) *Faulty Ruling of Charts.*—The rulings on the charts should be at right angles, or at the proper angle. The lines indicating a designated elongation may not be straight lines.

(b) *Faulty Cutting of Chart Sheets.*—The opposite edges of the chart sheet should be parallel and the sides should be at right angles to the top and bottom. The edges should be cut through a line printed outside the ruled area as a guide for this purpose. The presence of a cut line can be detected at the edge of a properly cut chart sheet.

(c) *Failure of Chart, Pen, and Clamp to Start Moving at the Same Time.*—Poor coordination of movement is indicated by a changing error in the recorded elongation when calculated on a percentage basis.

(d) *Improper Traverse Movement of the Pen.*—Improper traverse of the pen can be caused by a compacted cable or by a stretched cable on testing machines using this type of mechanism. It can also be caused by an error in the ratio of the circumference of two pulleys mounted on the same shaft.

(e) *Improper Magnification of Chart or Pen Movement.*—Magnification of the movement of the chart, where this is obtained by means of a movable pulley, will be in error if the diameter of the cable varies.

Section 7.—Change the first clause of the second sentence of the present Section 7 on clamps for fabrics to read as follows by the addition of the italicized words: "The dimension of all gripping surfaces parallel to the direction of application of the load shall be 1 in. *or more.*"

REVISION OF STANDARD, IMMEDIATE ADOPTION

Standard Methods of Testing and Tolerances for Knit Goods (D 231 – 39):

Section 13 (a).—Change to read as follows:

13. (a) The amount of grease shall be determined as follows: A representative specimen (Note) weighing about 5 g. shall be dried to constant weight in a ventilated oven at 105 to 110 C. It shall then be extracted with carbon tetrachloride for 4 hr. The extraction apparatus

shall be of a type wherein the carbon tetrachloride is siphoned from the extraction cup five times per hour, and siphoning shall not take place until the extraction cup contains not less than 125 ml. of carbon tetrachloride. The extract shall be dried to constant weight in a weighed flask at 105 to 110 C.

NOTE.—The bursting strength test specimens may be used for this test.

Figure 1.—Delete this figure.

EDITORIAL CHANGES IN STANDARDS

Standard Methods of Testing and Tolerances for Woolen Yarns (D 403 – 44):

Standard Methods of Testing and Tolerances for Worsted Yarns (D 404 – 44):

Section 10.—In both of these methods add the following note under this section:

NOTE.—The above moisture regain shall apply to woolen yarns sold commercially, but shall not apply to woolen hand knitting yarns sold at retail. In agreement with the proposed trade practice rules of the hand knitting industry, woolen hand knitting yarns are to be sold in the retail market on the basis of 11.1 per cent moisture regain, which is equivalent to 10 per cent moisture content as approved by the National Bureau of Standards. This basis shall be recognized for hand knitting yarn retail sales when the Federal Trade Commission promulgates the trade practice rules for the hand knitting yarn industry.

REPORT OF COMMITTEE D-14* ON ADHESIVES

Committee D-14 on Adhesives and its subcommittees held a three-day meeting in Atlantic City, N. J., October 9, 10, and 11, 1945, at which important progress was reported in various aspects of the committee's activities and at which time also a Symposium on Adhesives[1] was held. A second meeting of the committee and its subcommittees was held in Atlantic City on March 4 and 5, 1946.

At the present time, Committee D-14 consists of 85 members, of whom 39 are classified as producers, 33 as consumers, and 13 as general interest members. At present, there are 14 alternates for company and individual members.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, R. C. Platow.
Vice-Chairman, Henry Grinsfelder.
Secretary, L. P. Hart, Jr.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Strength Properties (M. H. Bigelow, chairman, A. G. H. Dietz, acting chairman) has been particularly active and has attracted the most interest and cooperation of the members. Mr. Dietz, who has been acting as chairman of this subcommittee during Colonel Bigelow's tour of duty in the European Theater of Operations, has been doing yeomen service in his temporary capacity.

The Section on Tensile Strength has reviewed the proposed new Tentative Method of Test for Tensile Properties of Adhesives. This method has been approved by Committee D-14 and subsequent to the Annual Meeting it will be submitted to the Administrative Committee on Standards for publication as tentative.[2]

The Section on Shear Strength has prepared the following three methods: Tentative Method of Test for Strength Properties of Adhesives in Plywood-Type Construction in Shear by Tension Loading, Tentative Method of Test for Strength Properties of Adhesives in Shear by Compression Loading, and Tentative Recommended Practice for Testing the Strength Properties of Adhesives.[2] These methods have been approved by the subcommittee and are now out to letter ballot of Committee D-14.

The Section on Impact Strength has prepared a test method which has been approved by the subcommittee and is now about ready to submit the method to the main committee. The Section on Fatigue Strength has started a round-robin research study to determine which of five suggested methods is the most reliable. The Section on Flexural Strength is hard at work trying to develop a method satisfactory to the subcommittee. The Section on Peel and Cleavage has prepared a method for determining the peel resistance of adhesives which will be submitted to letter ballot of the main committee after obtaining the approval of the subcommittee.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

[1] Symposium on Adhesives, Am. Soc. Testing Mats. (1946). (Symposium issued as separate publication.)

[2] See Editorial Note, p. 440.

Subcommittee II on Analytical Tests (L. Repsher, chairman) has been awaiting requests for methods from other committees. Work is under way on solids analysis.

Subcommittee III on Permanence (F. J. Wehmer, chairman) is another group that has aroused a maximum of interest and cooperation. Methods for measuring the effect of moisture and temperature are under study by the section. A method for determining the effect of chemical factors is now the subject of letter ballot in the main committee. Also a method for determining the effect of artificial and natural light on the permanence of adhesives is out for letter ballot of the committee. A comprehensive study of test methods for measuring the effect of biological factors on the strength of adhesives has been made and results of this study have been circulated to the subcommittee.

Subcommittee IV on Working Properties (C. B. Hemming, chairman) has developed and approved a method for determining the spreading rate which method is now out to letter ballot of the committee. Considerable work and discussion have taken place regarding test methods for cure, flow, slippage, tack, consistency, and blocking.

Subcommittee V on Specifications (G. Reinsmith, chairman) is making a serious effort to simplify the classification of adhesives. A questionnaire on the subject has been circulated to the committee.

Subcommittee VI on Nomenclature and Definitions (G. M. Kline, chairman) has submitted two lists of the more important terms for approval.

Plans are under way to have a committee meeting as well as subcommittee and advisory committee meetings at the Annual Meeting in June. A guest speaker, Dr. Woldemar A. Weyl (Director of Glass Science, Inc., State College, Pa.), will address the committee on the topic "The Theoretical Basis of Adhesion"[3] at that time.

This report has been submitted to letter ballot of the committee, which consists of 85 members; 61 members returned their ballots, of whom 59 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

R. C. PLATOW,
Chairman

HENRY GRINSFELDER,
Secretary.

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[3] See p. 1506.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-14 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of Test for:
Resistance of Adhesive Bonds to Chemical Reagents (D 896 - 46 T), and
Tensile Properties of Adhesives (D 897 - 46 T).

These recommendations were accepted by the Standards Committee on September 9, 1946, and the tentative methods appear in the 1946 Book of A.S.T.M. Standards, Part III-B.

On December 5, 1946 the Standards Committee accepted the Tentative Method of Test for Peel or Stripping Strength of Adhesives (D 903 - 46 T). The new tentative appears in the 1946 Book of A.S.T.M. Standards, Part III-B.

The proposed Tentative Recommended Practice for Determining the Effect of Artificial (Carbon-Arc Type) and Natural Light on the Permanence of Adhesives was accepted as tentative by the Standards Committee on December 31, 1946. The new tentative recommended practice has been assigned the designation D 904 – 46 T, and appears in the 1946 Book of A.S.T.M. Standards, Part III-B.

Committee D-14 submitted to the Standards Committee a proposed Introduction to the D-14 strength methods. This recommendation was accepted by the Standards Committee on November 23, 1946, and appears in the 1946 Book of A.S.T.M. Standards, Part III-B, as a preface to the adhesive strength methods D 897 and D 903.

On February 24, 1947, the Standards Committee accepted the following recommendations:

Tentative Method of:

Test for Determining the Applied Weight Per Unit Area of Dried Adhesive Solids (D 898 – 47 T),
Test for Determining the Applied Weight Per Unit Area of Liquid Adhesives (D 899 – 47 T),
Testing the Strength Properties of Adhesives in Shear by Compression Loading (D 905 – 47 T), and
Testing the Strength Properties of Adhesives in Plywood Type Construction in Shear by Tension Loading (D 906 – 47 T).

These new tentatives have been issued separately in reprint form.

REPORT OF COMMITTEE D-16*

ON

INDUSTRIAL AROMATIC HYDROCARBONS

There were no meetings during the past year for the general membership of Committee D-16 on Industrial Aromatic Hydrocarbons. Progress in the committee's work, however, was made possible through actions taken by the Advisory Committee at meetings held June 8, 1945, January 8 and April 8, 1946.

J. N. Roche has replaced D. F. Gould as chairman of Subcommittee I on Methods of Test for Crude Aromatic Products and E. C. Medcalf has succeeded R. F. Ruthruff as chairman of Subcommittee IV on Specifications for Aromatic Solvents due to the latter's resignation. With the discontinuance of Subcommittee I, J. N. Roche has been appointed chairman of Subcommittee V to replace J. B. Hill who recently resigned. A Subcommittee VI on Editorial Matters has been formed with E. T. Scafe as chairman.

I. NEW TENTATIVE METHOD

Inasmuch as it was the consensus of Committee D-16 that the procedure in Tentative Methods of Test for Specific Gravity, Color, and Hydrogen Sulfide and Sulfur Dioxide Content (Qualitative) of Industrial Aromatic Hydrocarbons (D 853 – 45 T)[1] dealing with specific gravity was inadequate for the purpose intended and actual details for conducting this test should be made available, the committee recommends for publication the Tentative Method of Test for Specific Gravity of Industrial Aromatic Hydrocarbons, as appended hereto.[2] In view of the development of this new method, the test for specific gravity will be omitted from Method D 853.

This recommendation has been submitted to letter ballot of the committee which consists of 33 voting members; 22 members returned their ballots, of whom 20 have voted affirmatively, 1 negatively, and 1 member marked his ballot "not voting."

II. REVISION OF TENTATIVE

Tentative Specifications for Industrial Grade Benzene (D 836 – 45 T):[1]—The committee recommends the following change in these specifications:

Section 2.—Add the following to the requirements: "Acidity. . . . No free acid, that is no evidence of acidity."

This recommendation has been submitted to letter ballot of the committee which consists of 33 voting members; 19 members returned their ballots, of whom 18 have voted affirmatively, 0 negatively, and 1 member marked his ballot "not voting."

III. EDITORIAL CHANGES IN TENTATIVES

The committee recommends that Tentative Methods D 848 and D 850 be editorially revised as follows:

Tentative Method of Test for Acid Wash Color of Benzene, Toluene, Xylenes and

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] 1945 Supplement to Book of A.S.T.M. Standards, Part III.

[2] This method was accepted as tentative by the Society and appears in the 1946 Book of A.S.T.M. Standards, Part III-A.

Similar Industrial Aromatic Hydrocarbons (**D 848 – 45 T**):[1]

Title.—Insert "Refined Solvent Naphthas" after "Xylenes."

Tentative Method of Test for Distillation of Industrial Aromatic Hydrocarbons (**D 850 – 45 T**):[1]

Section 5(a).—In line 25, insert "the" between the words "of" and "rise." In the same paragraph, line 26, insert "the" after the word "from."

Section 5 (b).—In line 7, change "distillate" to read "sample." In the same paragraph, line 16, insert "the" after the word "at."

Section 6 (b).—Change the description of "*K*" in the legend to read as follows: "*K* = Correction factor for the thermometer = 0.000154 = difference between coefficients of expansion for glass and for mercury." In the same legend, change the description of "*T*" to read as follows: "*T* = observed reading of the thermometer in degrees Centigrade, and."

Section 6 (d).—In line 11, change "values of" to read "formulas for."

Table I.—In the title, change "values of" to read "formulas for."

Delete the second column of this table and center the third column so that each formula involving "*k*" and "*P*" will be on a single line.

Change the footnote to this table to read as follows: "[a] Corrected barometric pressure in millimeters of mercury at 0 C. at time and place of distillation test = "P" in formulas for "k."

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Methods of Test for Crude Aromatic Products (J. N. Roche, chairman).—This subcommittee has had in preparation for publication as tentative a method of test for refined water-white constituents in light oil. The question was raised as to whether this subject falls within the scope of Committee D-16 activities and agreement has been reached that it does not. Therefore, Subcommittee I has been discharged.

Subcommittee II on Methods of Test for Refined Aromatic Products (V. J. Altieri, chairman).—A method of sampling industrial aromatic hydrocarbons has been prepared for subcommittee approval and recommendation for publication as tentative.

In connection with the Tentative Method of Test for Paraffins in Industrial Aromatic Hydrocarbons (D 851 – 45 T) and the Tentative Method of Test for Solidifying Point of Benzene (D 852 – 45 T), due cognizance is being taken of work being done respectively by Subcommittee XXV on Analysis of Petroleum Products for Hydrocarbon Types of Committee D-2 on Petroleum Products and Lubricants, which is now working on a procedure for determining the saturated hydrocarbon content of aromatic concentrates, and Section A on Thermometers, of Committee E-1 on Methods of Testing which is actively working on specifications for a solidifying point thermometer.

Arrangements have been made for the cooperative testing of 5 aromatic hydrocarbons (1 benzene, 2 toluenes, and 2 xylenes) to determine the degree of usefulness of the tentative methods submitted by Committee D-16 last year.[1] Members of Committees D-2 and D-1 on Paint, Varnish, Lacquer, and Related Products as well as D-16 are cooperating in these tests which are now under way.

Methods of test for the determination of sulfur content and of thiophene in benzene and toluene are now under study. Consideration is also being given to the development of a single phase method for acidity that is to replace or act as alternate for the present Tentative Method D 847.

The present method for specific gravity not being considered adequate, this subcommittee prepared for publication as tentative the more detailed methods for conducting this test for aromatic products including hydrometer, pycnometer, and Westphal balance procedures referred to earlier in this report.

Some fundamental studies on copper corrosion methods directed toward the development of a quantitative method for measuring the amount of sulfide corrosion are being undertaken by this group and a cooperative test program planned to determine its practicability.

Subcommittee III on Specifications for Aromatic Chemicals (J. N. Wickert, chairman).—A study by this subcommittee has indicated a possible need for specifications covering thiophene-free benzene and thiophene-free toluene but it is not sufficient to justify submittal of specifications for approval at present.

Subcommittee V on Nomenclature (J. Bennett Hill, chairman).—This group has considered the suggestion that the use of the term "naphtha" be eliminated from such names as solvent naphtha and similar products because of possible confusion as to the character of the material. The majority of the committee believe these terms are so well established that an attempt to change the official designation should not be made at this time. It was suggested that any producer who desires to do so may eliminate the name "naphtha" from his brand name.

Special Committee on *Volume Temperature Corrections.*—

Mr. W. H. Fulweiler was requested to consider and recommend means for the development of coefficient of expansion tables for aromatic hydrocarbons. A study which he made of presently available information bearing on this subject was presented in a report to the Advisory Committee at its meeting on June 8, 1946. This memorandum covers the status of the three Standard Volumetric Correction Tables (D 206, D 347, and D 633) now available in Society publications, the means at the Society's disposal to prepare the coefficient of expansion of aromatic hydrocarbons data requested by Committee D-16, and the recommendation that the A.S.T.M. should assume the responsibility of the development of these volume correction tables. The latter was made inasmuch as it is a logical development of the A.S.T.M. program of standardization and that the work should be done as an A.S.T.M. research project under the guidance of a representative committee, utilizing the scientific services of industry manpower with possibly some financial assistance from the Research Fund of the Society. It was Mr. Fulweiler's belief that the successful completion of such a project should reflect considerable credit on the Society and that a program to this end could be consummated with a minimum of delay.

A meeting of the Coordinating Group comprising representatives of Committee D-16 and Committee D-1 on Paint, Varnish, Lacquer, and Related Products held a meeting on June 26, 1946. The various points on which the overlapping specifications of these two committees are at variance were discussed individually and it may be that there are no insurmountable difficulties in the way of ultimately making both sets of specifications and test methods identical.

In general, the points of difference are the distillation procedure and four requirements (non-volatile, odor, water, and mercury corrosion) which are not now in the D-16 standards.

Both committees appear to have their specifications and methods of test under active review and the joint coordinating group thus finds itself in a favorable position to make progress.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, D. F. Gould.

Vice-Chairman, V. J. Altieri.

Secretary, W. L. Douthett.

This report has been submitted to letter ballot of the committee, which consists of 33 voting members; 25 members returned their ballots, of whom 24 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. M. WEISS,
Chairman.

W. L. DOUTHETT,
Secretary.

REPORT OF COMMITTEE D-17*

ON

NAVAL STORES

The last regular meeting of Committee D-17 on Naval Stores was held on June 27, 1944, during the time of the 1944 Annual Meeting of the Society at New York. The widely separated headquarters' locations of the committee membership and difficulties attending travel during the past year made it inadvisable to try to hold a meeting of the committee. The officers and subcommittee chairmen have been active in the development of test procedures, and several new members have been added to the committee. The committee lost one of its original and most active and valuable members through the resignation of C. E. Kinney, formerly the voting representative of Hercules Powder Co., who was retired by his company. Mr. Kinney had until a year ago been chairman for many years of Subcommittee I on Softening Point of Rosin, and his duties in this capacity have been taken over by J. M. Schantz, who has been designated the voting representative of Hercules Powder Co. The chairman of Subcommittee VIII on Terpene Hydrocarbons and Pine Oil, I. E. Knapp, was forced to relinquish that position due to a change of employment, and this chairmanship has been taken over by R. E. Price, Chief Chemist, Crosby Chemicals, Inc., Picayune, Miss. This firm is a large producer of a diversified line of naval stores products obtained from pine stumpwood.

I. NEW TENTATIVES

The committee recommends for publication as tentative the following two new methods:

Tentative Methods of:

Test for Water in Liquid Naval Stores[1], and
Test for Volatile Oil in Rosin.[1]

II. REVISIONS OF TENTATIVES

The committee recommends the following revisions of four tentatives:

Definitions of Terms Relating to Naval Stores and Related Products (**D 804 - 45 T**),[2] revised as appended hereto.[3]

Tentative Methods of Sampling and Testing Pine Tars and Pine-Tar Oils (**D 856 - 45 T**):[2]

Section 6.—Change to read as follows:

6. (*a*) Specific gravity of pine tar and pine-tar oil shall be expressed as the ratio of the weight of a given volume of the material at 25 C. (77 F.) to that of an equal volume of water at 15.5 C. (60 F.) and shall be reported to the third decimal place as:

Specific gravity, 25/15.5 C. (77/60 F.)....

(*b*) The specific gravity may be determined at any convenient room temperature with a 25-ml. Hubbard pycnometer or weighing bottle as described in the Standard Method of Test for Specific Gravity of Road Oils, Road Tars, Asphalt Cements, and Soft Tar Pitches (A.S.T.M. Designation: D 70)[4] being sure to use the weight of water contained by the pycnometer at the temperature of the test. Since the specific gravity of pine tar and pine-tar oil at room temperature changes by 0.0005 per degree Centigrade change in temperature, add or subtract 0.0005 for each degree that the temperature is, respectively, above or below 25 C. Finally convert such corrected value to the 25/15.5 C. basis by multiplying by the factor 0.998.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

[1] These methods were accepted as tentative by the Society and appear in the 1946 Book of A.S.T.M. Standards Part II.

[2] 1945 Supplement to Book of A.S.T.M. Standards, Part III.

[3] These revised definitions were accepted by the Society and appear in the 1946 Book of A.S.T.M. Standards, Part II.

[4] 1946 Book of A.S.T.M. Standards, Part II.

(c) As an alternate procedure, the specific gravity may be determined (somewhat less accurately) with a suitable hydrometer, such as that described in the Standard Method of Test for Specific Gravity of Creosote (A.S.T.M. Designation: D 368).[4] If the temperature of the tar is above or below 25 C., correct the reading by means of the same value for change in specific gravity, that is 0.0005 per degree Centigrade.

NOTE.—For very heavy viscous tars it may be advantageous to warm the sample to 40 to 45 C., in which case the correction factor for correcting the observed reading of the hydrometer to 25 C. will be 0.0004 per degree Centigrade.

Section 13.—Change the formula to read as follows:

Absolute viscosity (centipoises) = $2.13 \times t \times d \times f$

Change the definition of "*f*" to read as follows:

f = a correction factor for the instrument. If a standard oil is used for calibration, having its viscosity stated in seconds for a standard instrument, this factor is equal to the ratio of standard seconds to observed seconds for the instrument used. If the viscosity is stated in centipoises, this value, with the observed time and the density of the oil shall be substituted in the above formula in order to calculate *f* for the instrument. The value of *f* should be calculated for both 30 C. and 50 C.

NOTE 1.—Change to read as follows:

While the true Herschel formula for petroleum oils at 50 C, is $C = \left(2.13\,t - \frac{160}{t}\right) \times d \times f$, experiments with pine tars indicate that results within the limit of experimental error, close enough for practical purposes, and in good agreement with results obtained with the Stormer viscosimeter, may be expected by the use of the simpler formula given above.

Section 14 (b).—Change the second and third sentences to read as follows:

When the maximum temperature to be read or stated in a specification is indicated by the thermometer, or as soon as a drop in temperature occurs before the maximum specified temperature is reached, discontinue the heating, allow the condenser to drain, and record the total quantity distilled to that temperature. Record the maximum temperature reached during the test if it is below the maximum temperature specified in the test.

Section 20.—Change to read as follows:

20. *Acid Number.*—In a small porcelain crucible or glass weighing capsule, accurately weigh 0.2 to 0.3 g. of sample. Place 100 ml. of alcohol (Formula 30 or other laboratory grade) in a 250-ml. wide-mouth Erlenmeyer flask, and add a small quantity of Alkali Blue (dry powder) from the end of a small spatula. The alcohol should take on a distinct blue color. Add 0.1 *N* NaOH dropwise until the color just changes from blue to red. Introduce the crucible and sample and bring into solution by warming on a hot plate. A blue-green color will again develop. If the blue is masked by the color of the tar, add additional indicator. Titrate with the NaOH until no blue remains in the color of the solution when the flask is held at an angle against a white background.

Tentative Methods of Sampling and Testing Dipentene (D 801 – 44 T):[5]

Section 7.—Change to read as follows:

7. Determine the specific gravity at 15.5/15.5 C. by any convenient method, reporting the value to the nearest 0.0005. Determinations made at any other temperature using apparatus standardized at 15.5 C. shall be corrected by adding or subtracting from the observed reading 0.00082 for each degree Centigrade that the temperature of the liquid is above or below 15.5 C.

Section 14.—Change to read as follows and add a new Paragraph (*b*):

14. (*a*) Transfer 5 ml. of the sample to a 100-ml. glass-stoppered graduate. Add V. M. and P. naphtha or mineral spirits (Note) in successive 5-ml. portions shaking well after each addition. Both the sample and the petroleum solvent shall be at the same temperature, 20 to 25 C., when used. Any appreciable proportion of dissolved moisture will be indicated by the development of turbidity in the mixture. Record the quantity of petroleum solvent added when turbidity develops. Test a reference sample in the same manner and compare.

NOTE.—The petroleum solvent should be redistilled before being used in the test, discarding the first and last 10 per cent fractions.

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[5] 1944 Book of A.S.T.M. Standards, Part II.

(*b*) ***Alternate method.***—If a quantitative moisture determination is indicated by the above test, or is otherwise required, follow the procedure described in the Tentative Method of Test for Water in Liquid Naval Stores (A.S.T.M. Designation: D 890).[4]

New Section.—Add a new Section 15 to read as follows:

NOTE.—If desired, the aniline and mixed aniline points of dipentene may be determined as described in the Tentative Method of Test for Aniline Point and Mixed Aniline Point of Petroleum Products (A.S.T.M. Designation: D 611),[4] instead of in accordance with the following Section 15.

15. *Aniline Point and Mixed Aniline Point:* (*a*) *Apparatus.*—The apparatus shall consist of the following:

(*1*) *Test Tube.*—A test tube approximately 18 ml. in capacity, 15 mm. in diameter, and 125 mm. in length, with a lip, and fitted with a two-hole cork stopper, one for the thermometer and the other a 2-mm. hole for the stirrer.

(*2*) *Stirrer.*—A stiff copper wire, approximately 1 mm. in diameter, coiled at the lower end (3 to 5 coils). The coils shall be of such diameter that the stirrer can move freely up and down around the thermometer. The upper end of the wire stirrer shall extend several inches through the 2-mm. hole in the cork stopper with the end of the wire looped or bent to form a handle.

(*3*) *Thermometer.*—An A.S.T.M. Aniline Point Thermometer, graduated in Centigrade degrees, having a range of -38 to $+42$ C., and conforming to the requirements for thermometer 33C – 41 T as prescribed in the Tentative Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1).[4]

(*4*) *Bath.*—A 300-ml. tall-form beaker or other convenient receptacle containing a suitable clear nonaqueous medium, such as turpentine, kerosine, etc., for use as a cooling or heating bath.

(*b*) *Reagents:* (*1*) *Aniline.*—Dry c.p. aniline over KOH and redistill, discarding the first and last 10 per cent of the distillate. Preserve the purified aniline in small amber-colored glass-stoppered bottles. Suitable aniline should be practically colorless.

(*2*) *Normal Heptane.*—Normal heptane for use in the mixed aniline point test shall have a boiling point in the range of 98 to 98.5 C. The *n*-heptane shall be dried before use by shaking for several minutes with anhydrous Na_2SO_4 and filtering to obtain a clear, dry liquid.

(*c*) *Procedure for Aniline Point.*—Clean and dry the apparatus. Pipette into the test tube 5 ml. of the sample, previously dried in accordance with the procedure for drying *n*-heptane (Paragraph (*b*)). By means of a safety pipette[6] or small burette fitted with a calcium chloride tube, add an equal quantity of aniline. Stopper the test tube with the cork containing the thermometer and the stirrer and mix the contents thoroughly with the wire stirrer. Clamp the test tube in a suitable position to permit raising and lowering of the beaker containing the prechilled cooling medium. (A temperature of -15 to -20 C. may be required.) Immerse the test tube in the bath and stir the mixture rapidly, avoiding the inclusion of air bubbles. While stirring constantly, record the temperature at which the solution becomes cloudy or turbid throughout (Note). Allow the solution to warm up until cloud disappears and repeat the cooling process again recording the temperature at which the solution becomes cloudy. The average of the temperatures at which the solution becomes cloudy throughout shall be reported as the aniline point. These two readings should agree within 0.5 C.

NOTE.—The true aniline point is characterized by a turbidity which increases sharply as the temperature is lowered.

(*d*) ***Procedure for Mixed Aniline Point.***—Pipette 2.5 ml. of the sample, 2.5 ml. of *n*-heptane and 5 ml. of aniline[7] into the test tube. Proceed as described in Paragraph (*c*), except that the solution may have to be warmed slightly above room temperature to produce a clear solution. To do this immerse the test tube in a warm bath and stir constantly until the solution is clear. Remove the test tube from the bath, and allow the contents to cool while stirring constantly. Record the temperature at which the solution becomes cloudy throughout. Repeat the procedure and report the average temperature as the mixed aniline point. The readings should agree within 0.5 C.

Tentative Methods of Sampling and Testing Pine Oils (**D 802 – 44 T**):[5]

Section 5.—In the first sentence delete "having a precision of 0.0005" and substitute the following: "reporting the value to the nearest 0.0005. (A pycnometer or specific gravity balance is recommended; a hydrometer should not be used, on account of errors caused by surface tension on the hydrometer stem.)"

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[6] Aniline is poisonous and should not be pipetted by direct application of the mouth to the pipette, nor should any be allowed to remain on the hands, even in *very small quantities*, as aniline will be absorbed through the skin.

Section 9.—Letter the present section as Paragraph (*a*) and add a new Paragraph (*b*) to read as follows:

(*b*) *Alternate Method.*—Determine the moisture content of pine oil according to the procedure described in the Tentative Method of Test for Water in Liquid Naval Stores (A.S.T.M. Designation: D 890).[1]

Section 10 (*a*).—In item (*1*), change the capacity of the flask from "250-ml." to read "500-ml." Change item (*3*) to read as follows: "A straight tube, water-cooled glass, reflux type, having a water jacket 400 mm. ($15\frac{3}{4}$ in.) in length, with inner tube approximately 10 to 12 mm. in diameter."

Section 10 (*b*).—Add the following note to item (*3*): "NOTE.—If dipentene is used, it shall have been treated according to this procedure, to remove traces of terpene alcohol that may be present."

The recommendations appearing in this report have been submitted to letter ballot of the committee, which consists of 40 voting members, with the results shown in Table I.

III. REAFFIRMATION OF STANDARD

The committee recommends that the Standard Method of Test for Toluene Insoluble Solid Matter in Rosin (Chiefly Sand, Chips, Dirt, and Bark) (D 269 - 30) be reaffirmed since this standard has stood for more than six years without revision.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Softening Point of Rosin (J. M. Schantz, chairman).—The former chairman of the subcommittee, C. E. Kinney, over the course of several years, collected and reviewed a large number of different test methods used or proposed for measuring the softening point of rosin and similar resins. The present chairman has continued to work on this project and has compiled a rather comprehensive abstract of some 25 different test procedures and modifications which the committee will study. The abstracts cover descriptions of the bath or heat-transfer system, essential apparatus, preparation of sample, and salient features of the methods. It is planned to have a careful study and discussion of the various methods, at a meeting of Committee D-17, from which it is hoped that it will be possible to select several methods for further intensive collaborative study, with the view to standardization. The results of a study made in one of the collaborating laboratories of a modification of the Tentative Method of Test for Softening Point (Ball and Shouldered Ring Apparatus) (E 28 - 42 T) were published. This modification is based on heating of the sample in a small air chamber, surrounded by another air bath maintained at a constant temperature, with the temperature selected so as to have a definite relation, within narrow limits, to the expected or observed softening point temperature. Reprints of the paper are available from the chairman of Committee D-17.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVES			
Test for Water in Liquid Naval Stores	26	0	3
Test for Volatile Oil in Rosin	26	0	3
II. REVISIONS OF TENTATIVES			
Def. of Terms Relating to Naval Stores and Related Products (D 804 - 45 T)	27	0	2
Sampling and Testing Pine Tars and Pine-Tar Oils (D 856 - 45 T)	27	0	2
Sampling and Testing Dipentene (D 801 -44 T)	27	0	2
Sampling and Testing Pine Oil (D 802 - 44 T)	27	0	2
III. REAFFIRMATION OF STANDARD			
Test for Toluene Insoluble Solid Matter in Rosin (Chiefly Sand, Chips, Dirt, and Bark) (D 269 - 30)	20	0	3

Subcommittee III on Volatile Oils in Rosin (W. C. Smith, chairman).—The chairman of the subcommittee, in connection with his official duties in a government laboratory, made a study of several methods used to separate residual volatile oils from rosin. With the assistance of a member of the staff of another laboratory engaged in similar work, additional tests were made of a series of rosins containing added known quantities of naturally occurring residual volatile oil (such as higher boiling turpentine fractions). Samples were also prepared with added quantities of certain oils of petroleum origin, such as are used in the extraction of rosin from wood, and from turpentine still wastes in the recovery of so-called "reclaimed" rosin. Low softening point and other objectionable properties result from insufficient removal of volatile oil from rosin, and occasionally rosins are put on the market with too high a volatile oil content. The method is based on a gradual removal of the oil by steam distillation in the presence of glycerine, with a constantly increasing temperature obtained by reduction of the ratio of water to glycerine in the apparatus. The results were reviewed by several committee members and the preparation for further collaborative study was recommended of a proposed tentative method submitted herewith.[1]

Subcommittee IV on Chemical Analysis of Rosin (W. D. Pohle, chairman).—This subcommittee has continued its study of acid and saponification methods for dark-colored rosins and of a proposed method for unsaponifiable matter in rosin, which was originally submitted with the 1944 report[7] of the committee. The results of collaborative tests so far available do not seem to warrant recommendation at this time with regard to either the present Tentative Method of Test for Acid Number of Dark Rosin (D 465 - 45 T) or the proposed method for unsaponifiable matter in rosin. However, it is hoped to report further progress along this line in the near future. One of the results brought to light by the collaborative testing was a nonuniformity of the color change characteristics of the indicator used in the tentative method, known as Clayton yellow.

Subcommittee V on Tall Oil (A. Pollak, chairman).—The chairman has submitted to the subcommittee a complete tabulation of all data reported by collaborators who tested the samples sent out in 1945, in order that the variations in the reported results could be more carefully compared with the details of the various test procedures. One of the principal points brought to light was an apparent tendency to obtain an appreciably higher percentage of rosin acids in tall oil when using the modified Wolff method, as compared with the results by the alternate McNicol method. Modifications of both of these test methods are being studied in detail in the laboratory of the subcommittee chairman. A full discussion of all these collaborative test data is planned for the annual meeting of the committee, from which it is hoped to be able to decide what modifications are indicated and desired for better coordination of the analytical results by the two alternative methods. Further collaborative study will then be undertaken.

The subcommittee has approved certain editorial changes in the Tentative Methods of Testing Tall Oil (D 803 - 44 T) so as to refer to all standardized test reagents in terms of normality in place of the less common reference to molal strength.

Subcommittee VI on Pine Tar and Tar Oil (J. P. Bain, chairman).—Collaborative tests have been made by a number of the subcommittee members on samples of pine tar and pine tar oil produced by the retort process, and a sample of pine tar obtained by the "kiln" process of burning, using the procedures covered by the Tentative Methods of Sampling and Testing Pine Tars and Pine-Tar Oils (D 856 - 45 T). From a careful study of the test methods and the collaborative data received from five laboratories, it is concluded that good reproducibility can be had and expected from most of the individual procedures. The distillation test did not give as close checks as might be desired, and some additional study will be given to this procedure. The subcommittee, as a result of this study, recommends revisions in Methods D 856 as covered earlier in this report.

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[7] *Proceedings*, Am. Soc. Testing Mats., Vol. 44, p. 495 (1944).

Subcommittee VII on Terpene Hydrocarbons and Pine Oil (R. E. Price, chairman).—The results of the collaborative tests on samples of dipentene and pine oil sent out in 1945 have been further studied by the chairman. From these results, and some additional methods that have been recommended for inclusion, the subcommittee submitted the revisions in Tentative Methods D 801 as covered earlier in this report.

The presence of moisture in solution or in suspension often affects the usefulness of certain kinds of naval stores products such as dipentene, pinene, and pine oil. The subcommittee has studied the results obtained and published by a member of the committee using a modification of the so-called Karl Fischer method and recommends the adoption of a new tentative method, based on the published data, for the quantitative determination of water in liquid naval stores products.[1]

Subcommittee IX on Definitions (J. L. Boyer, chairman).—The tentative definitions relating to naval stores have received careful study by the subcommittee during the past year. Many of the definitions have been revised, and a number of new ones have been added. The definitions that have been modified include those for abietic acid, Archangel pitch, brewer's pitch, Burgundy pitch, limed rosin, modified rosin, reclaimed rosin, and terpene alcohol. The terms for which definitions are submitted for the first time include the following: colophony, metallic resinates, and rosin type (sample). These new and revised definitions are appended hereto[1] as a revision of Tentative Definitions D 804.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, V. E. Grotlisch.
Vice-Chairman, J. L. Boyer.
Secretary, W. A. Kirklin.
Members-at-Large of Advisory Committee: R. P. Chapman, I. E. Knapp, W. D. Pohle, A. Pollak.

This report has been submitted to letter ballot of the committee, which consists of 40 voting members; 29 members returned their ballots, of whom 28 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

V. E. GROTLISCH,
Chairman.

W. A. KIRKLIN,
Secretary.

REPORT OF COMMITTEE D-18*
ON
SOILS FOR ENGINEERING PURPOSES

Committee D-18 on Soils for Engineering Purposes has held one meeting during the past year, on March 1, 1946, in Pittsburgh, Pa.

On November 19, 1945, the committee lost a valuable member by the death of Prof. F. C. Lang, of the University of Minnesota and the Minnesota Highway Department. Prof. Lang had been chairman of Subcommittee V on Stabilized Soils and later headed the Symposium Committee in preparing for a General Symposium on Soil Test Procedures. A joint resolution of Committee C-9 on Concrete and Concrete Aggregates, Committee D-4 on Road and Paving Materials and Committee D-18 memorializing Professor Lang has been incorporated in the March 1, 1946, minutes of the committee.

A number of members have returned from service with the Armed Forces and are resuming their work with the committee. Prof. W. S. Housel is one of those returning to civilian life, and he has consented to take over the chairmanship of the Symposium Committee left vacant by the death of Prof. Lang.

Subsequent to the 1945 Annual Meeting, Committee D-18 presented to the Society through the Administrative Committee on Standards a proposed Tentative Method of Test for Specific Gravity of Soils. This recommendation was accepted[1] by the Standards Committee on June 27, 1945.

At a meeting of the Joint Subcommittee of Committees D-4 and D-18 on Tests and Specifications for Stabilized Soils, a paper entitled "Development of a Method of Test for Evaluating Soil Bituminous Mixtures"[2] by E. O. Rhodes and P. F. Phelan, chairman and secretary of the Bituminous Producers Coöperative Research Committee was presented by Mr. Phelan. This method will be submitted to the membership of the Joint Subcommittee for consideration for publication as tentative.

Prof. D. M. Burmister presented a paper on "The Aims and Objectives of Committee D-18" at the meeting held June 26, 1946, at Buffalo. This paper is being recommended for publication in the ASTM BULLETIN.[3]

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, E. J. Kilcawley.
Vice-Chairman, W. S. Housel.
Secretary, K. F. Vernon.
Members-at-Large of Advisory Committee: H. F. Clemmer, K. B. Woods, M. D. Catton, D. M. Burmister.

ACTIVITIES OF SUBCOMMITTEES

Reports of subcommittee chairmen made at the June, 1946, meeting of Committee D-18 indicated renewed interest in committee affairs. These reports likewise indicated that there will be considerable activity during the coming year in all subcommittees.

Subcommittee I on Nomenclature and Definitions (W. P. Kimball, chairman) recommends that the Tentative Definitions of Terms and Symbols Relating to Soil Mechanics (D 653 - 42 T) be retained as tentative. The subcommittee plans to review these definitions in the near future and to develop a list of definitions of soil mechanics terms.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

[1] In submitting this recommendation to the Standards Committee, Committee D 18 reported the following results of the letter ballot vote from a committee membership of 80, 58 members returned their ballots, 53 of whom voted affirmatively, 3 negatively and 2 members marked their ballots "not voting."

[2] This paper was subsequently presented at the 1946 Annual Meeting and appears on p. 1416.

[3] ASTM BULLETIN, No. 142, October, 1946, p. 38.

Subcommittee III on Methods of Sampling (C. W. Allen, acting chairman) plans to investigate possible applications of seismic methods of determining character of foundation strata and use of aerial photographs in soil maps.

Subcommittee IV on Methods of Testing the Physical Characteristics of Soils (Harold Allen, chairman) during the coming year, plans to revise the following methods: Standard Methods of Preparing Soil Samples for Mechanical Analysis and Subgrade Soil Constants (D 421 – 39), Mechanical Analysis of Soils (D 422 – 39), and Test for Liquid Limit (D 423 – 39).

This subcommittee also recommended that the following four standards which have been published for six years without revision be continued in their present form: Standard Methods of Test for: Plastic Limit and Plasticity Index of Soils (D 424 – 39), Centrifuge Moisture Equivalent (D 425 – 39), Field Moisture Equivalent (D 426 – 39), and Shrinkage Factors of Soils (D 427 – 39).

The subcommittee recommends that the Tentative Method of Test for Moisture-Density Relations of Soils (D 698 – 42 T) be continued as tentative with a revision which has been approved by letter ballot of the subcommittee and will be submitted to Committee D-18.

A proposed new Tentative Method of Test for Field Density of Soil in Place was approved by letter ballot of Subcommittee IV and will be submitted to Committee D-18.

Subcommittee VI on Methods of Testing for Compressibility and Elasticity of Soils (D. M. Burmister, chairman) plans to work on proposed new methods of consolidation tests.

Subcommittee VII on Methods of Testing for Shearing Resistance of Soils (F. J. Converse, chairman) at a meeting on March 1, 1945, received a report on results of cooperative shear tests on Ottawa sand. This report showed a wide divergence in results obtained and indicated that a great deal of research is needed. The subcommittee plans to prepare a method of direct shear test.

Subcommittee VIII on Methods of Testing Soil Stability under Triaxial Loading (R. F. Blanks, chairman) will proceed with reorganization of the subcommittee according to the plan approved by the Advisory Committee. With reorganization completed, the subcommittee plans to go ahead with the research phases of triaxial shear testing.

Subcommittee X on Methods of Testing for Bearing Capacity of Piles (*Load Tests*) (A. E. Cummings, chairman) has plans for continuing work on methods of testing for which this subcommittee is responsible.

Subcommittee XI on Methods of Testing Drainage Properties of Soils (*Capillarity and Permeability*) (K. B. Woods, chairman) has in mind the organization of several sections to work on the different aspects of the problems involved in developing methods of tests for measuring permeability and capillary rise of water in soils.

Joint Subcommittee of Committees D-4 and D-18 on Tests and Specifications for Stabilized Soils (H. F. Clemmer, chairman).—A program has been set up that will entail considerable work in this subcommittee during the coming year.

This report has been submitted to letter ballot of the committee, which consists of 80 voting members; 51 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

E. J. KILCAWLEY,
Acting Chairman.

K. F. VERNON,
Acting Secretary.

REPORT OF COMMITTEE D-19*

ON

WATER FOR INDUSTRIAL USES

Committee D-19 on Water for Industrial Uses and its subcommittees held meetings at Pittsburgh, Pa., on February 19 and 20, 1946. Two regularly scheduled meetings were not held during 1945, due to travel restrictions. The Advisory Subcommittee held a meeting on October 21, 1945.

F. N. Speller, and Max Hecht, the Society's representatives on the Executive Subcommittee, Joint Research Committee on Boiler Feedwater Studies, presented no report because no meetings were held during 1945.

Max Hecht, the Society's representative to the Feedwater Treatment Committee, Boiler Code Committee, American Society of Mechanical Engineers, offers no report due to the fact that no meetings were held during 1945. Work has continued during 1945 on proposed revisions of the Rules on Inspection of Material and Steam Boilers comprising Section VI, Part II, of the A.S.M.E. Boiler Construction Code. The working committee expects to report the draft of these revisions to the Feedwater Treatment Committee later this year.

A chapter on Boiler Corrosion was submitted to and accepted by the Editorial Committee of the Electrochemical Society for inclusion in its Handbook on Corrosion. This chapter, submitted on behalf of Committee D-19, was prepared under the joint authorship of E. P. Partridge, W. C. Schroeder, S. F. Whirl, and Max Hecht, all members of the committee.

The present officers of the committee have been reelected for the ensuing term of two years.

I. NEW TENTATIVE METHODS

Tentative Methods of Test for Dissolved Oxygen in Industrial Waters.[1]—These methods were proposed as tentative in 1945, following approval by letter ballot of Subcommittee IV on Methods of Analysis, the sponsoring subcommittee, and Committee D-19. The results of the letter ballot vote in the subcommittee from a membership of 52, were as follows: affirmative 35, negative 2, "not voting" 7. The results of the letter ballot vote in Committee D-19 from a membership of 73 were as follows: affirmative 31, negative 4, "not voting" 23. The recommendation for these proposed tentative methods was presented to the Administrative Committee on Standards on August 1, 1945, but was not accepted, due to the fact that more than 10 per cent of the votes cast were negative and because the methods had not been reviewed at a regular meeting of Committee D-19. On the recommendation of the Standards Committee these methods, which include one referee and three nonreferee procedures, were published as information in the 1945 Report of Committee D-19,[2] and also were republished with a review statement in the ASTM BULLETIN.[3] The action taken at the D-19 Advisory Committee meeting on October 21, 1945, authorized the chairman to present the proposed methods at the next regular meeting of Committee D-19. The methods were accordingly reviewed at the meeting of the committee on February 19, 1946. At this time all accumulated communications were presented, including suggestions from both affirmative and negative voters and comments secured by the publication of the methods in the December, 1945, ASTM BULLETIN. No new and additional data were contributed during the meeting, and accordingly no major change in the printed methods was deemed necessary. The members present voted that certain editorial changes be incorporated and that the methods be resubmitted for acceptance, subject to letter ballot vote. The results of this letter ballot vote of Committee D-19, March 21, 1946, from a membership of 78 was as follows: 56 affirmative, 2 negative, and 12½ "not voting." It was desirable to revise Section 5 (*i*) of these methods. The results of the letter ballot vote of Committee D-19, May 29, 1946, are shown in Table I.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] This method was accepted as tentative by the Society and appears in the 1946 Book of A.S.T.M. Standards, Part III-A.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 390 (1945).

[3] ASTM BULLETIN, No. 137, December, 1945, p. 19.

Tentative Recommended Practice for Field Sampling of Water-Formed Deposits.—The committee recommends that this recommended practice as prepared by Section B of Subcommittee III on Methods of Sampling be accepted for publication as tentative, as appended hereto.[1]

II. REVISION OF TENTATIVE METHOD

Tentative Method of Field Test for Tendency of Boiler Water to Cause Embrittlement Cracking of Steel (**D 807 – 44 T**).[4]—The following changes prepared by Subcommittee VI on Methods of Testing are recommended for inclusion in this method:

Section 3 (*a*).—Add the number 1 after the word "Note" at the end of this Paragraph and make the same addition in the reference to it in the fourth sentence of this paragraph.

Section 3 (*c*).—Add the following new Note 2 at the end of this paragraph. Insert a reference to it at the end of the second sentence.

NOTE 2.—The surface to be studied is the stressed area, which starts ¼ in. above the spot corresponding to the opening in the test block and extends about 1 in. toward the adjusting screw.

Fig. 3.—Add the following end view of the test specimen shown in the accompanying Fig. 1.

Base Block.—Add the detailed dimensions of the base block shown in the accompanying Fig. 2.

Section 4 (*b*).—In the third sentence change "a more continuously concentrated solution" to read "an increasingly concentrated solution."

Section 5.—In the first sentence of Paragraph (*b*), change the reference to Standard Specifications A 70 to read "Standard Specifications for Carbon-Silicon Steel Plates of Ordinary Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels (A.S.T.M. Designation: A 201)."

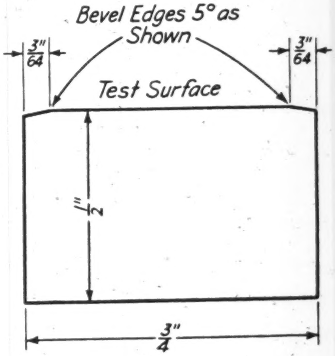

FIG. 1.—End View of Test Specimen (Adjusting Screw Hole Not Shown).

Add a new Paragraph (*c*) to this section to read as follows:

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[4] 1944 Book of A.S.T.M. Standards, Part III.

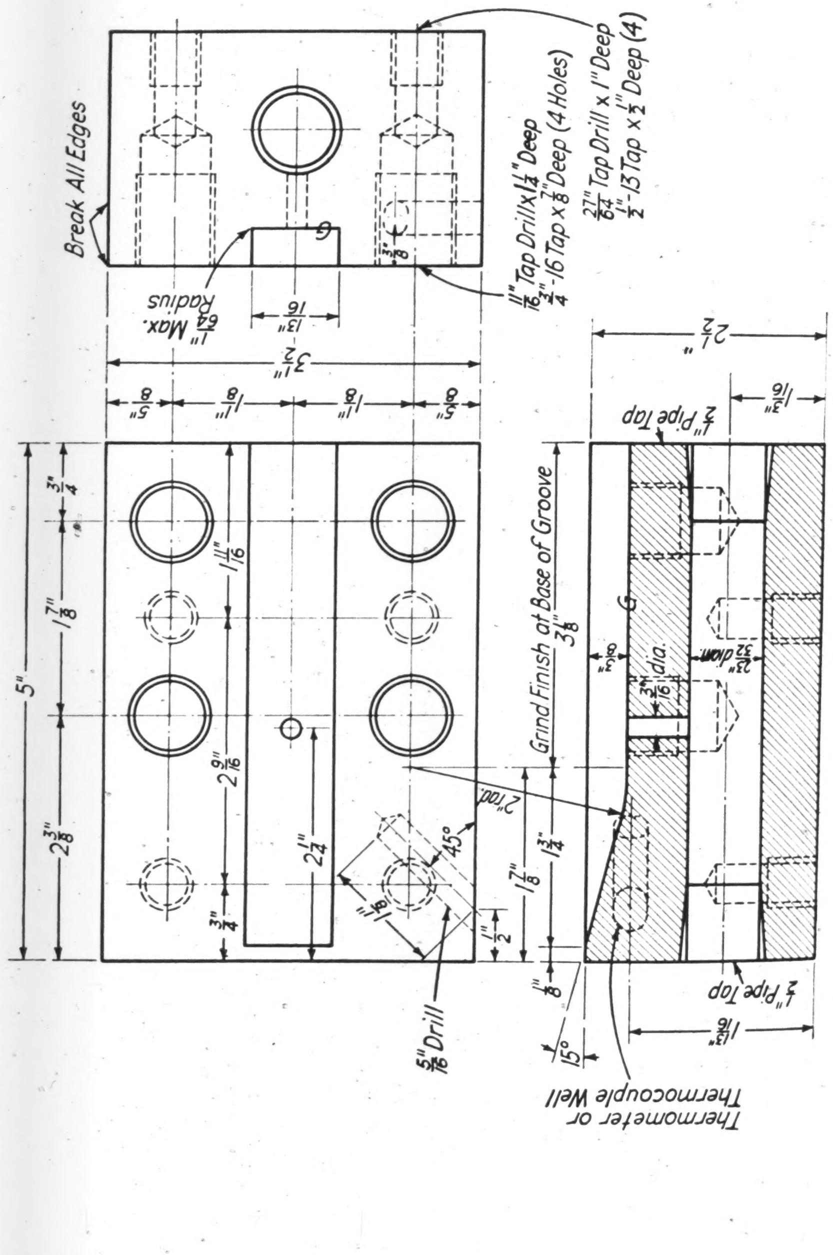
Break All Edges
11/16" Tap Drill x 1 1/4" Deep
3/4"-16 Tap x 7/8" Deep (4 Holes)
27/64" Tap Drill x 1" Deep
1/2"-13 Tap x 1/2" Deep (4)
1/64" Max. Radius
13/16"
3 1/2"
5/8"
1 1/8"
1 1/8"
5/8"
3/8"
G
5"
3/4"
1 7/8"
2 3/8"
1 11/16"
2 9/16"
3/4"
2 1/4"
45°
1 1/16"
1/2"
2" rad.
5/16" Drill
Grind Finish at Base of Groove
3 3/8"
1 3/4"
1 7/8"
1/8"
15°
Thermometer or Thermocouple Well
1/4" Pipe Tap
1/4" Pipe Tap
2 1/2"
1 3/16"
1 3/16"
3/16" dia.
23/32" diam.
G

FIG. 2.—Base Block.

(*c*) Alloy steels are often more susceptible to cracking than the standard cold-rolled steel specified for test specimens. Where the water tested is used in alloy-steel boilers, it is desirable that the test specimen be prepared from the same material or from bars of similar composition and physical properties.

Section 6.—Add the following as a new Paragraph (*b*), renumbering the present Paragraph (*b*) as (*c*):

(*b*) The test surface shall be given a 5-deg. bevel as shown in Fig. 3.

Section 8 (*b*).—In the second sentence change "drilled into the block" to read "provided for it in the block."

Section 9 (*g*).—Add the following as a new Note 2, renumbering the present Note 2 as 3. Change the present reference to "Note 2" to read "Note 3," and add a reference to Note 2 at the end of the first sentence.

NOTE 2.—The one-sixth of a turn of the adjusting screw is a maximum. Approach it cautiously and do not repeat if the haze does not appear.

Section 9 (*i*).—In the second sentence, change "may" to read "will."

Section 9 (*j*).—In the first line, delete the word "desired."

Section 11(*c*).—In Note 3, change "sometimes distorts" to read "may distort."

Section 11(*d*).—Add the following as a new Note 5 to this section, inserting a reference to it after the word "embrittlement" at the end of the paragraph:

NOTE 5.—Embrittlement cracks in alloy steels often tend to be intracrystalline, following slip planes rather than grain boundaries.

III. REVISION OF STANDARD, IMMEDIATE ADOPTION

Standard Method of Reporting Results of Analysis of Industrial Waters (**D 596 – 41**).[4]—As proposed by Subcommittee V on Classifications, Committee D-19 recommends the immediate adoption of the following revisions. The committee accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society:

Section 2.—Add the following new definitions and Notes to this section as items (*g*) and (*h*):

(*g*) ***Alkalinity.***—Alkalinity is the capacity of a water for neutralizing acid.

NOTE 1.—Alkalinity is measured by the amount of standard acid which must be added to change the pH value of the water to some arbitrary value, as determined by the color change of an indicator or by an instrument.

NOTE 2.—Alkalinity is expressed fundamentally in terms of chemical equivalents of hydrogen ion required to neutralize the water to the specified reference point. It has commonly been expressed in terms of amount of calcium carbonate which would neutralize the required amount of acid to the specified reference point or points.

(*h*) *Hardness.*—Hardness is the capacity of a water for precipitating soap.

NOTE 1.—Soap is precipitated chiefly by calcium and magnesium ions commonly present in industrial water but may also be precipitated by ions of other polyvalent metals, such as iron, manganese, and aluminum and by hydrogen ions.

NOTE 2.—For industrial waters, hardness is customarily measured by the amount of soap required to produce a stable lather. Measurement is usually made on water sample, the alkalinity of which has been adjusted to the phenolphthalein end point to eliminate the effect of hydrogen ions.

NOTE 3.—Hardness is expressed fundamentally in terms of the chemical equivalents of metal ion capable of precipitating soap. It has commonly been expressed in terms of the equivalent amount of calcium carbonate.

Table I.—Delete the footnote reference mark "‡" from the item "pH, electrometric" under the heading "Miscellaneous."

IV. DEFINITION PUBLISHED AS INFORMATION

Definition of Distilled Water (*Published as information only*).—Committee D-2 on Petroleum Products and Lubricants requested Committee D-19 to furnish either a specification or definition for distilled water, for use in chemical testing. Members present at the February 19, 1946, meeting formulated the following definition and authorized its publication as information only:

> *Distilled Water.*—Distilled water may be defined as water containing not more than 0.5 ppm. of dissolved solids, having a specific conductivity not greater than 2 micromhos per centimeter at 25 C. and a pH between 6.5 and 7.5 at 25 C.
>
> NOTE.—For specific applications, different limits may be required or permissible but water of the above defined quality is satisfactory for a great majority of applications requiring distilled water. Water satisfying the requirements of the above definition when freshly distilled may have a pH as low as 5.5 after equilibrium with the atmosphere is reached.

The recommendations in this report have been submitted to letter ballot of the committee, which consists of 73 members, with the results shown in Table I.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
NEW TENTATIVE METHODS			
Test for Dissolved Oxygen in Industrial Waters	54½	1	7½
Recommended Practice for Field Sampling of Water-Formed Deposits	58	0	5
REVISION OF TENTATIVE			
Test for Tendency of Boiler Water to Cause Embrittlement Cracking of Steel (D 807 - 44 T)	53½	0	8½
REVISION OF STANDARD, IMMEDIATE ADOPTION			
Method of Reporting Results of Analysis of Industrial Waters (D 596 - 41)	57	1½	4½

ACTIVITIES OF SUBCOMMITTEES

Mr. O. M. Elliott of the Sun Oil Co., Philadelphia, Pa., was afforded an opportunity to make a preliminary presentation of his colorimetric procedure for the determination of dissolved oxygen at a meeting of the Advisory Subcommittee held in Pittsburgh on October 21, 1945. The following is his abstract of the method:

> This report to A.S.T.M. Committee D-19 discusses preliminary work on a colorimetric test for dissolved oxygen in low concentrations. Refinements to the Winkler method are outlined. To date, all continue to use a titration or back titrations. A colorimetric test for dissolved oxygen in low concentrations is possible with the absorption indicator alpha-napthoflavon. This indicator is more sensitive to iodine than starch. Its iodine absorption characteristics are outlined.
>
> A detailed laboratory procedure for a Winkler reaction colorimetric test for dissolved oxygen in low concentrations is outlined. Briefly, this consists of iodizing portions of the blank with varying known amounts of iodate; then adding sample and blank portions to alpha-napthoflavon by flash mixing technique and matching colors.
>
> The indicator sensitivity is estimated to be plus or minus 0.001 ml. per liter and plus or minus 0.0015 ml. per liter with free iodine equivalent to dissolved oxygen content of 0.005 and 0.010 ml. per liter respectively. Above 0.010 ml. per liter the flash mixing technique becomes critical. Fixing reagents must be reduced to 1 ml. each and HCl used in place of H_2SO_4.
>
> No claims are made as to absolute accuracy.

Subcommittee II, Editorial (E. P. Partridge, chairman).—The objective and general outline of the projected Manual on Industrial Waters was reviewed in detail. The Subcommittees on Sampling, Analysis, Classification, and Methods of Testing will be in a position to contribute material that has been published in the 1944 Book of A.S.T.M. Standards, Part III and its 1945 Supplement.

Other chapters of the Manual would be devoted to uses of water, composition, sampling, analysis, and identification of water-formed deposits. Definitions published by the committee will be included in a glossary. A chapter on the properties of water was suggested, as well as a chapter on the interpretation of analytical results.

Subcommittee III on Methods of Sampling (D. K. French, chairman).—The

Subject Subsection on Sampling of Stationary Boilers of Section A on Water is preparing revisions to the Tentative Recommended Practice for Sampling Boiler Water from Stationary Boilers (D 860 – 44 T).

Subject Subsection on Sampling of Water at Atmospheric Pressure has made a study during 1945 for proposed extensive revisions of the Standard Methods of Sampling Plant or Confined Waters for Industrial Uses (D 510 – 41) and will present these revisions at a subsequent meeting of the subcommittee for comment and suggestions.

Section B on Water-Formed Deposits intends to study more detailed sampling procedures which it will offer for suggestions and comments to the subcommittee as proposed revisions to the field sampling method being recommended as tentative this year.

Section C on Steam was organized at the February 19, 1946, meeting to work on the development of methods for sampling steam.

Subcommittee IV on Methods of Analysis (A. A. Berk, chairman).—The Subject Subsection on Iron, Aluminum, and Manganese under Section A on Analytical Methods has been studying revisions to the Tentative Method of Test for Total Aluminum and Aluminum Ion in Industrial Waters (D 857 – 45 T) and for Manganese in Industrial Waters (D 858 – 45 T). The proposed revisions will be offered to the subcommittee at a subsequent meeting for suggestions and comments. Four procedures are under study for determining iron and when agreement has been reached a proposed method will be submitted.

The Subject Subsection on Silica has been studying proposed revisions to Tentative Methods of Test for Silica in Industrial Waters (D 859 – 45 T). The proposed revisions will be offered to the subcommittee at a subsequent meeting for suggestions and comments.

Progress reports were offered at the February 19, 1946, meeting by the following subject subsections: Alkali Metals in Water, Dissolved Solids, Nitrogenous Substances, and Oil in Water.

The members approved the appointment of the personnel of a subject subsection to undertake revisions to Standard Method for Determination of Total Carbon Dioxide and Calculation of the Carbonate and Bicarbonate Ions in Industrial Waters (D 513 – 41). The major revisions concern themselves with the apparatus feature of this method. If practical, nonreferee methods are to be prepared.

Section B on Electrometric Methods reported that work on pH methods would be deferred until after the Symposium on pH Measurement to be held at the 1946 Annual Meeting of the Society. The work on conductivity and polarographic methods remains to be organized.

Section C on Special Methods reported that the work discussed in the 1945 Annual Report[5] of the committee was being continued. The question of nomenclature is of considerable importance and the desirability of using chemical names, mineral terms, or a combination of mineral terms with oxide designations was stressed. A report of this latter activity will be offered at a subsequent meeting of the subcommittee.

Subcommittee V on Classification (L. K. Herndon, chairman).—At the February 20, 1946 meeting L. K. Herndon was elected as chairman of this subcommittee and M. D. Baker as secretary.

J. A. Holmes, the retiring chairman, had developed during 1945 definitions for alkalinity and hardness, and for terms used in water-formed deposits, which appear in the sampling method being submitted as tentative. Standard Method D 596 is being revised as noted elsewhere in this report.

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[5] *Procedings*, Am. Soc. Testing Mats., Vol. 45, p. 356 (1945).

Section B on Reporting Analyses of Water-Formed Deposits submitted a preliminary draft on a proposed method for reporting the results of analysis of water-formed deposits at the February 20, 1946, meeting of the committee. This method, for the present, is directed to reporting the results of the chemical analysis, and eventually it will provide for reporting results by microscopic, spectrographic, and X-ray diffraction methods. The preliminary draft has been circulated to the members of the subcommittee for comment and suggestions at a subsequent meeting.

Subcommittee VI on Methods of Testing (V. V. Kendall, chairman).—Under Section A on Corrosivity, the subject subsection on the Embrittlement Tester offered proposed revisions to Tentative Method D 807 as listed earlier in this report.

The subject subsection on the N. D. H. A. Corrosion Tester submitted a revision to the third draft of this method. The paragraphs of the method have been changed to make the method all inclusive, so that it can be used for measuring the corrosion rates in any water supply. The draft is being circulated to the subcommittee members for suggestions and comments and will be reconsidered at a subsequent meeting of the committee.

This report has been submitted to letter ballot of the committee, which consists of 81 members; 63 members returned their ballots, of whom 58½ have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

MAX HECHT,
Chairman.

R. E. HALL,
Secretary.

REPORT OF COMMITTEE D-20*

ON

PLASTICS

Since the June, 1945, meeting in New York, N. Y., Committee D-20 on Plastics and its subcommittees have held two meetings in Atlantic City, N. J. in October, 1945, and in March, 1946.

L. A. Sontag has been appointed chairman of Subcommittee IX to succeed W. L. Hess. M. E. Marks has been appointed chairman of Subcommittee I to succeed Lt. Col. M. H. Bigelow. H. K. Nason has been appointed to represent Committee D-20 on Sectional Committee Z26 on Safety Glass. W. N. Findley has been recommended for appointment on Committee E-9 on Fatigue.

Special committees have been appointed on the subjects of Simulated Service Testing with E. F. Seaman, chairman, and Ultimate Consumer Goods with Ephraim Freedman, chairman. These committees are to study their respective fields for the purpose of determining how Committee D-20 can be of most service.

A special committee on Prosthetics with W. E. Gloor as chairman has been appointed to work with the Thomas England General Hospital on problems relative to artificial limbs, and similar devices.

A special committee representing Committee D-9 on Electrical Insulating Materials and Committee D-20 with J. D. Ryan as chairman has been appointed to assist the armed forces in the technologic problems arising in connection with the proposed research program on plastics at Princeton University.

Subsequent to the 1945 Annual Meeting, Committee D-20 presented to the Society through the Administrative Committee on Standards a number of recommendations which are listed in Table I together with the results of the letter ballot vote in Committee D-20 and the date of acceptance by the Committee on Standards. The new and revised tentatives have been given the designations indicated and appear in the 1945 Supplement to Book of A.S.T.M. Standards, Part III.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is submitting four proposed new tentatives, revisions in ten tentatives, revisions in one standard for immediate adoption, and recommends the adoption as standard of three tentatives. The titles and designations of these items are listed in Table II and the several revisions recommended are given in detail in the Appendix.

These recommendations have been submitted to letter ballot of the committee, which consists of 138 voting members, with the results shown in Table II.

ACTIVITIES OF SUBCOMMITTEES

All of the subcommittees have been quite active. The work on specifications has been somewhat lessened while that on test methods, analytical procedures, and definitions has increased.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

At the present time all of the subcommittees are engaged in writing statements on the significance of the tests for which they are responsible.

Subcommittee I on Strength Properties (M. E. Marks, chairman).—Work is being continued on the influence of the span-depth ratio on test results in flexural testing and on the influence of column length in compression testing. Various methods of evaluating impact strength and fatigue properties are also under study. Tentative methods for determining shear strength and bearing strength will soon be submitted to vote in the subcommittee. A new section on the bonding strength of laminates has been set up. Other items under consideration are a mandrel type flexibility test and methods for evaluating the strength properties of various types of laminates including high- and low-density materials and glass-fiber types.

TABLE I.—RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS, INCLUDING RECORD OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
New Tentative Methods of Test for:			
Designating the Flow Temperature for Thermoplastic Molding Materials (D 863 – 45 T), *accepted January 17, 1946*	35	2	31
Coefficient of Cubical Thermal Expansion of Plastics (D 864 – 45 T), *accepted December 10, 1945*	47	1	18
Revision of Tentatize Methods of Test for:			
Deformation of Plastics Under Load (D 621 – 45 T), *accepted December 10, 1945*	43	1	24
Revision of Tentative Specifications for:			
Phenolic Molding Compounds (D 700 – 45 T), *accepted December 10, 1945*	31	3	42

Subcommittee II on Hardness Properties (L. Boor, chairman).—Studies are being continued on various methods of evaluating hardness properties, such as indentation, scratch, and falling abrasion methods. The Rockwell method is also being studied further with the hope of broadening its scope and increasing its accuracy.

TABLE II.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVES			
Test for Estimating Blocking of Sheet Materials	44	1	30
Test for Tensile Properties of Thin Plastic Sheets and Films	56	0	25
Test for Deviation of Line of Sight Through Transparent Plastics	43	0	39
Definitions of Terms Relating to Plastics:			
Plastic, as an adjective	64	0	17
Pseudo-stable	62	3	16
II. REVISIONS OF TENTATIVES			
Spec. for Cellulose Acetate Molding Compounds (D 706 – 44 T)	40	1	40
Spec. for Cellulose Acetate Butyrate Molding Compounds (D 707 – 44 T)	38	1	42
Spec. for Nonrigid Vinyl Chloride-Acetate Resin Plastics (D 742 – 44 T)	40	1	40
Spec. for Ethyl Cellulose Molding Compounds (D 787 – 44 T)	40	1	40
Spec. for Cellulose Nitrate (Pyroxylin) Plastic Sheets, Rods, and Tubes (D 701 – 44 T)	26	0	55
Test for Impact Resistance of Plastics (D 256 – 43 T)[a]	63	1	17
Test for Tensile Properties of Plastics (D 638 – 44 T)	63	0	18
Recommended Practice for Long-Time Tension Tests of Plastics (D 674 – 42 T)	51	2	28
Rec. Prac. for Resistance of Plastics to Accelerated Service Conditions (D 756 – 44 T)	53	0	28
Method of Conditioning Plastics (D 618 – 45 T)	55	2	24
III. REVISION OF STANDARD, IMMEDIATE ADOPTION			
Method of Test for Acetone Extraction (D 494 – 41)[b]	48	2	31
IV. ADOPTION OF TENTATIVES AS STANDARD			
Spec. for Cast Methacrylate Plastic Sheets, Rods, Tubes, and Shapes (D 702 – 43 T)	32	0	49
Spec. for Urea-Formaldehyde Molding Compounds (D 705 – 43 T)	33	0	48
Test for Shear Strength of Plastics (D 732 – 43 T)	47	1	33

[a] The recommended revision of Method D 256 was submitted subject to concurrence of Committee D-9 but this revision was not approved by Committee D-9 in time for adoption in 1946.

[b] The recommended revision of Method D 494 was submitted subject to concurrence of Committee D-9 and was approved by that committee, see p. 407.

Subcommittee III on Thermal Properties (W. A. Zinzow, chairman).—A round-robin test together with a questionnaire involving the flow temperature of thermoplastic materials is under way. It is hoped to find ways and means of revising the method to give more reliable results. Work is also under way in an attempt to broaden the scope of the heat distortion method and on developing a satisfactory method for determining ignition temperatures of plastics.

Subcommittee IV on Optical Properties (W. F. Bartoe, chairman).—Development of methods of test involving the use of the General Electric recording spectrophotometer and the use of an integrating sphere are being continued. Further work is also being done on the method for haze and light transmission.

Subcommittee V on Permanence Properties (J. H. Teeple, chairman).—The Tentative Recommended Practice for Operating Light and Water Exposure Apparatus (D 822 – 45 T),[1] prepared by Committee D-1 on Paint, Varnish, Lacquer and Related Products, is being studied in order to determine its applicability to plastics. Studies on weight loss on heating by use of the Abderhalden type oven are being continued, and a tentative method should soon be ready for subcommittee letter ballot. The method for water vapor permeability is under consideration with the idea of bringing it in line with similar methods developed by other groups. Also under consideration are revisions of the colorfastness test and the accelerated weathering tests; it is hoped that controlled voltage to the light source will greatly improve these methods.

Subcommittee VI on Specifications (G. M. Kline, chairman).—Work on tentative specifications for polystyrene sheets, plates, rods and tubes is being continued. The subcommittee is continuing the cooperative work with the National Electrical Manufacturers Association on tentative specifications for post-forming laminating materials. A study is being made on the possibility of combining the specifications for vinylidene chloride (D 729) and vinyl chloride-acetate resins (D 728). This subcommittee is continuing its cooperation with the Society of the Plastics Industry on a Classification of Plastics.

Subcommittee VII on Analytical Properties (P. D. Brossman, chairman).—Methods for determining free phenol and formaldehyde in phenolic resins and insolubles in polystyrene resins are being developed. Work is being continued on a method for determining plasticizer, residual solvent, pigment, and filler in cellulose ester plastics. A revision of the Tentative Method of Test for Specific Gravity (D 792 – 44 T) has been sent to letter ballot in the subcommittee.

Subcommittee VIII on Research (H. K. Nason, chairman).—The program on impact properties is being continued and a formal program on flow properties is being set up. Some developments of this subcommittee are being incorporated in the existing test methods and others will be ready for submission in the near future. Papers of an original nature are solicited for future meetings.

Subcommittee IX on Molds and Molding Processes (L. A. Sontag, chairman).—Problems under active consideration are the effects of molding and mold design on physical properties, standardization of mold design of test specimens, and standardization of molding technique. Several items have been submitted to letter ballot in the subcommittee.

Subcommittee X on Definitions, Nomenclature, and Significance of Tests (G. W. Clark, chairman).—A number of definitions are under study. A standard system for designating the direction of testing specimens is being developed. Considerable work is under way on the subject of significance of tests; as the other subcommittees propose statements on the significance of the tests under their jurisdiction, they will submit them to this subcommittee for final consideration before being presented to Committee D-20.

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[1] 1945 Supplement to Book of A.S.T.M. Standards, Part II.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, Robert Burns.
First Vice-Chairman, G. M. Kline.
Second Vice-Chairman, M. L. Macht.
Secretary, A. J. Warner.

This report has been submitted to letter ballot of the committee, which consists of 138 voting members; 85 members returned their ballots, of whom 76 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

ROBERT BURNS,
Chairman

L. W. A. MEYER,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-20 presented to the Society through the Administrative Committee on Standards the proposed Tentative Definition of the Noun Plastic. This recommendation was accepted by the Standards Committee on July 10, 1946, and the new tentative appears in the 1946 Book of A.S.T.M. Standards, Part III-B bearing the designation D 883 - 46 T.

On September 9, 1946, the Standards Committee accepted the following recommendations submitted by Committee D-20:

Revision of Tentative Specifications for:

Cellulose Acetate Molding Compounds (D 706 - 46 T),
Laminated Thermosetting Materials (D 709 - 46 T),
Cellulose Acetate Plastic Sheets (D 786 - 46 T), and
Ethyl Cellulose Molding Powders (D 787 - 46 T).

The revised tentative specifications appear in the 1946 Book of A.S.T.M. Standards, Part III-B.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR PLASTICS

In this Appendix are given recommendations affecting certain standards and tentatives covering plastics which are referred to earlier in this report.[1] These standards and tentatives appear in their present form in the 1944 Book of A.S.T.M. Standards, Part III.

REVISIONS OF TENTATIVES

Tentative Specifications for Cellulose Acetate Molding Compounds (D 706 – 44 T):

Tentative Specifications for Cellulose Acetate Butyrate Molding Compounds (D 707 – 44 T):

Tentative Specifications for Nonrigid Vinyl Chloride-Acetate Resin Plastics (D 742 – 44 T):

Tentative Specifications for Ethyl Cellulose Molding Compounds (D 787 – 45 T):

In each of the above four specifications it is recommended that the requirements for dielectric strength be deleted from Table II, and also that the reference to the dielectric strength test in Section 6 be omitted. It is generally agreed that this property is not critical in the case of these plastic molding compounds and hence its inclusion in these specifications is considered an unnecessary burden.

Tentative Specifications for Cellulose Nitrate (Pyroxylin) Plastic Sheets, Rods, and Tubes (D 701 – 44 T):

The following revision of these specifications is recommended in order to improve the analytical procedures:

Section 4.—Change the first sentence of Paragraph (*b*) to read as follows by the addition of the italicized words and the omission of those in brackets: "Cellulose nitrate plastic shall be manufactured from approved cellulose nitrate (Paragraph (*a*)) with suitable plasticizers, or from reworked material having *an acidity not greater than 25 epm.* (see Note 1, Section 6 (*d*)) and a fuming-off temperature of not less than [160 C. (320 F.)] *165 C. (329 F.)* (Section 6 (*c*)), or from mixtures of the two."

In Paragraph (*d*) change the fuming-off temperature from "160 C. (320 F.)" to read "165 C. (329 F.)."

Add the following new Paragraph (*e*) to read as follows, relettering the present Paragraph (*e*) as (*f*): "(*e*) *Acidity.*—The free acidity of the finished plastic shall not exceed 15 epm. (see Note 1, Section 6 (*d*))."

Section 6.—Change Paragraph (*b*) to read as follows:

(*b*) *Residual Volatile Matter.*—From the sample, cut shavings 0.005 to 0.010 in. in thickness, transferring these shavings immediately into a tared weighing bottle until approximately 1 g. of the sample has been prepared. Care shall be taken to keep evaporation of solvents at a minimum. Weigh the sample and the container to the nearest 0.1 mg. Heat with the cover removed in a vacuum oven at 50 C. (122 F.) under a vacuum of at least 28 in. of mercury for 2 hr. Cool, weigh, and return to the oven for another 2 hr. at 50 C. (122 F.). Cool and weigh again. The loss in weight during the second heating is assumed to be due to the loss of plasticizer, and loss of plasticizer is also assumed to take place during the first heating period at the same rate. Calculate the residual volatile matter as follows:

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[1] See p. 461.

$$\text{Residual volatile matter, per cent} = \frac{\text{first wt. loss} - \text{second wt. loss}}{\text{wt. of sample}} \times 100$$

It is recognized that there are other methods of test which are more efficient as control tests but which are of limited application and cannot be used in a general specification. In case any of these tests are used, the vacuum oven test for determining residual volatile matter, as set forth in this paragraph, shall be used as referee in case of dispute. It must be further recognized that the vacuum oven test provides relative data only; it does not measure the absolute value of the volatile constituents.

Change Paragraph (*c*) to read as follows:

(*c*) *Fuming-off Temperature.*—The sample shall be prepared by taking shavings not thicker than 0.001 in. from the test piece which shall be clean and free from finger-prints. The shavings shall be taken by scraping the plastic with a suitable scraper or with a freshly broken piece of glass. To free the shavings of excessive moisture and solvents they shall be heated for 2 hr. in an oven at 50 C. (122 F.). Approximately 0.1 g. of the blended shavings shall be introduced into a dry, clean, 6 by 0.5 in. test tube which shall then be *lightly* stoppered with a new perforated or notched cork stopper and immersed 2 in. in a liquid bath at 100 C. (212 F.). The bath shall be heated so that the temperature rises at the rate of not less than 3 nor more than 5 C. (5 and 9 F.) per min. until the sample decomposes explosively. This temperature shall be recorded as the fuming-off temperature. Not fewer than 5 tests shall be run on each sample, and the average of the five reported as the fuming-off temperature. The test shall be repeated for all samples which do not decompose explosively.

Replace the present Paragraph (*d*) on plasticizer content with the following:

(*d*) *Free Acid.*—A 1.00 ± 0.1-g. sample of the material shall be dissolved in 10 ml. of distilled water and 100 ml. of acetone. When solution is complete the cellulose nitrate and plasticizers shall be precipitated by adding another 25 ml. of water in such a manner that a fine slurry is formed. A few drops of methyl red indicator shall be added and the suspension titrated with 0.01 N sodium hydroxide. A blank shall be run on the acetone-water mixture. Calculate the amount of free acid as follows:

$$\text{Acidity, epm.} = \frac{(R - S)T}{W} \times 1000$$

where:

R = milliliters of NaOH solution required to titrate the sample,

S = milliliters of NaOH solution required to titrate the blank,

T = normality of the NaOH solution, and

W = grams of sample used.

NOTE 1.—Epm. is an abbreviation for equivalents per million. It is a useful measure for expressing acidity or alkalinity in terms of acidic hydrogen concentration in parts per million. Acidity expressed as epm. is independent of the kind of acid present.

NOTE 2.—In case a dye in the plastic obscures the methyl red end point, the end point may be taken at a pH of 5.3 in electrometric titration.

Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (D 256 – 43 T):

In order to introduce a standardized notching procedure in the preparation of specimens for the impact strength test, the following revision is recommended:

Section 3 (*a*).—Change the second sentence of this paragraph to read as follows: "To insure the correct contour and condition of the specified notch, all specimens shall be notched in accordance with the directions in Section 4."

New Section.—Add a new Section 4 to read as follows, renumbering the subsequent sections accordingly:

4. *Notching Test Specimens:* (*a*) Notching shall be done with a milling cutter. The cutting edge shall be carefully ground and honed to insure sharpness and freedom from nicks and burrs. The included cutting angle of the tool shall be 45 ± 1 deg. with a radius of 0.010 ± 0.001 in. at the intersection of the arms of this angle. Tools with no rake and with a work-relief angle of 15 to 20 deg. have been found satisfactory.

(*b*) The tool shall be so mounted that the plane which bisects the included cutting angle is perpendicular to the axis of rotation within plus or minus 2 deg. Any standard milling machine or lathe may be used for the cutting operation.

(*c*) Inspection of a notched specimen may be made instead of inspection of the cutter for any material in which the relation between notch dimensions and cutter dimensions has been established. The contour and alignment of multi-toothed cutters shall be determined from the notch which the cutter produces in a suitable material.

(*d*) The linear speed of the cutting tool shall not exceed 600 ft. per min. (Note 5).

(*e*) The feed shall be slow enough to result in a smooth cut. Speeds in the range from 0.5 to 5.0 in. per min. have proved satisfactory for some materials (Note 5).

NOTE 5.—While the speed ranges given in Paragraphs (*d*) and (*e*) have produced satisfactory notches in some plastic and electrical insulating materials, it may be necessary to study the effect of variation in these conditions when unfamiliar materials are to be notched.

(*f*) After each 500 notches, or more often, if hard abrasive materials are being notched, the cutter or a notched specimen shall be inspected for sharpness, freedom from nicks, radius of tip and angle. If any signs of dullness are apparent, or if the angle and radius do not fall within their specified limits the cutter shall be replaced with a newly sharpened and honed one. A microscope with a camera lucida attachment is suitable for checking the radius and angle of either the cutter or notch.

NOTE 6.—Relatively close tolerances must be imposed upon the angle and radius of the notch because these factors largely determine the degree of stress concentration at the base of the notch during the test. The maintenance of a sharp, clean-edged cutting tool is particularly important since minor defects at the base of the notch can cause large errors in test results.

Section 5.—Renumber the present Note 5 in this section as Note 7.

Section 8 (a).—Change the second sentence to read as follows: "To insure the correct contour and condition of the specified notch, all specimens shall be notched in accordance with the directions in Section 4."

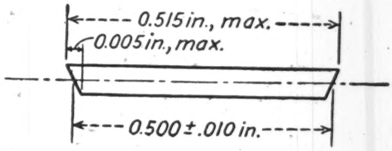
Tentative Method of Test for Tensile Properties of Plastics (D 638 – 44 T):

Fig. 1.—Change the tolerance in inches for dimension *W*-width of flat section, from "−0.000 and +0.005 in." to read "±0.010 in."

From the second sentence of footnote *a*, delete the phrase "suitably on a 210-in. minimum radius."

Add the following as footnote *e*, referenced at "W-Width of flat section" in the column "Dimension, in.":

[e] For molded specimens a draft of not over 0.005 in. may be allowed for the specimens 0.125 in. in thickness, and this should be taken into account when calculating width of the specimen. Thus, the cross-section of the molded specimen would be as follows:



Tentative Recommended Practice for Long-Time Tension Tests of Plastics (D 674 – 42 T):

The committee recommends that this recommended practice be made a method of test and revised as follows:

Title.—Change the words "Recommended Practice" to read "Method of Test."

Scope.—In the first line change the words "recommended practice" to read "method of test" and add the following new Paragraph (*c*): "(*c*) The engineering significance of data obtained by long-time tension tests is not presently known."

Section 4.—Delete this section on classifications of materials.

Section 7.—Replace this section with the following:

"7. *Conditioning Test Specimens.*—Test specimens shall be conditioned in accordance with the Tentative Methods for Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618)."

Section 8.—Delete Paragraph (*a*) and reletter the subsequent paragraph accordingly.

Section 10.—Change item (*6*) from "preconditioning procedure used" to read "conditioning procedure used." Omit item (*7*) and renumber the subsequent items accordingly.

Tentative Methods of Test for Resistance of Plastics to Accelerated Service Conditions (D 756 – 44 T):

The committee recommends a revision in the scope of this accelerated test method and also a few minor changes in the method of conditioning as follows:

Section 1.—Change the last sentence of Paragraph (*a*) from "(See Explanatory Notes 1 to 4)" to read "(See Explanatory Note 1)." In Paragraph (*b*) delete the reference to Note 3 in the first line.

New Section.—Add as a new Section 2 the present Explanatory Notes 1, 2, and 3, designated respectively as Paragraphs 2 (*a*), (*b*), and (*c*), and revised to read as follows:

2. (*a*) The testing conditions covered in these methods represent a start towards organizing a group of test procedures for determining the effects of specified changes of atmospheric temperature and humidity upon plastic articles. These procedures have been used for testing both thermosetting and thermoplastic materials.

NOTE.—The test procedures covered in these methods have been drawn from various tests used in branches of the plastics industry, and by government agencies in procurement of materials.

(*b*) While several of the testing temperatures are not used in other A.S.T.M. methods, they have a background of several years experience in portions of the plastics industry. Especially in procedures IV and V (Sections 11 and 12) the accelerating effect of temperature in prompting changes caused by a rise in humidity leads to temperatures which are well above those encountered in normal service, and they are used simply to accelerate change.

(*c*) Procedure I (Section 8) has been found to develop warping, weight change and exudation in plastic parts. Procedure II (Section 9) is designed to reveal poorly cured plastics by developing cracks in them. Procedures III and IV are more severe measures of the same tendencies developed in procedure I; the conditions of procedure IV will produce noticeable chemical decomposition in many plastics. Procedure V (Section 12) is especially valuable in testing the behavior of plastic parts with metallic inserts and laminates, for cracking on exposure to temperature change. Procedures VI and VII are modifications of procedure I, applying to impact-resistant and low heat-distortion temperature types of thermoplastics, respectively.

Section 4.—Renumber as Section 5 and change to read as follows:

5. *Conditioning.*—(*a*) Test specimens shall be conditioned prior to testing and reconditioned at the end of the test procedure at the standard laboratory atmosphere of 25 ± 1 C. (77 ± 2 F.) and 50 ± 2 per cent relative humidity, unless otherwise specified. Test specimens of material $\frac{1}{4}$ in. or less in thickness of plastic section shall be conditioned for a minimum period of 40 hr., and specimens of material over $\frac{1}{4}$ in. in section thickness shall be conditioned for a period of at least 88 hr. in accordance with the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).[2]

Explanatory Notes.—Delete the present Notes 1, 2 and 3. Insert the following four sentences after the first sentence of the present Note 4, renumbering it as Note 1: "In making such cycle tests, the initial conditioning shall be given, followed by exposure to the test as stated. Subsequent test procedures shall be applied without reconditioning the specimen at the end of each unit of the cycle. For instance, in carrying out a three-cycle exposure to procedure I, the first cycle shall be carried out in accordance with Sections 8 (*a*) to (*h*), the second cycle in accordance with Sections 8 (*c*) to (*h*), and the third cycle in accordance with Sections 8 (*c*) to (*j*). The reconditioning shall always be carried out at the end of the test cycle unless sample failure makes this step appear trivial."

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[2] 1946 Book of A.S.T.M. Standards, Part III-B.

Tentative Method for Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 45 T):

This method is being revised to include an additional functional procedure recommended for use when effects of exposure to heavier atmospheric moisture are to be determined. The revised method is appended hereto.[2]

REVISION OF STANDARD, IMMEDIATE ADOPTION

Standard Method of Test for Acetone Extraction of Phenolic Molded or Laminated Products (D 494 – 41):

New Section.—Add a new Section 2 to read as follows, renumbering the subsequent sections accordingly:

2. *Significance of Test.*—(*a*) For molded phenolic products, acetone extraction should be considered solely as a quantitative expression of a property normally associated with degree of cure. There is no demonstrably rigorous relation between the optimum mechanical and electrical properties of a well-cured piece and the numerical value of the acetone test. The amount of acetone-soluble matter is affected by: (*1*) Nature of resin and filler, (*2*) Lubricant, (*3*) Molding temperature, (*4*) Length of cure, (*5*) Thickness of the section from which sample is taken, (*6*) Nature of molded piece, (*7*) Technique used in molding, (*8*) Distribution of fines in the material to be extracted, and (*9*) Method of grinding specimen. These variations under some conditions may cause a difference of 3 to 4 per cent in acetone extractable matter. For this reason, the method should be used only as a comparative test for measuring undercure.

(*b*) For laminated phenolic products, acetone extraction indicates the change in stage of cure of the material, change in type of resin used, or presence of acetone extractable addition agents.

REPORT OF COMMITTEE E-1*
ON
METHODS OF TESTING

Committee E-1 on Methods of Testing has held no meetings since its previous report to the Society in 1944. A number of meetings have been held, however, of the various sections of the committee as noted later in this report. While many members of the committee found it necessary to devote their attention to war-time activities, it is now planned to review the programs of the various technical committees and it is expected that this will result in the formulation of an active program of work.

The Executive Committee of the Society appointed three representatives-at-large to serve on the Advisory Committee, namely, L. H. Fry, The Locomotive Institute, D. E. Parsons, National Bureau of Standards, and T. Smith Taylor, U. S. Testing Co., each for a three-year term expiring in 1948.

The committee records with sorrow the death on November 6, 1945, of Professor E. C. Bingham, one of its most active and cherished members; Professor Bingham was an authority and pioneer in the science of rheology. He rendered notable service to Committee E-1 for the past twenty years as chairman of Technical Committee II on Consistency, Plasticity, and Related Properties.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends revisions in two of its standards as given below, and asks for a nine-tenths affirmative vote at the annual meeting in order that these recommendations may be referred to letter ballot of the Society:

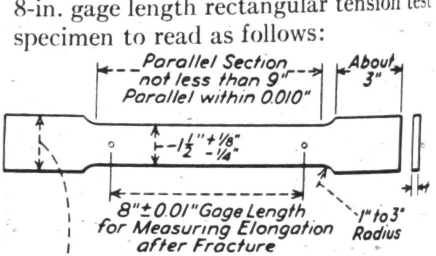
Standard Methods of Tension Testing of Metallic Materials (E 8 – 42):

Section 1.—Change the first sentence to read as follows by the addition of the italicized words: "These methods *and the figures which are a part of the methods* cover the apparatus, specimens, and procedures for the tension testing of metallic materials."

New Section.—Add the following new Section 4, renumbering the present sections accordingly:

4. *Test Specimens.*—Improperly prepared test specimens often cause unsatisfactory test results. It is important, therefore, that care be exercised in the preparation of specimens, particularly in the machining, to assure good workmanship.

Fig. 1.—Change the illustration of the 8-in. gage length rectangular tension test specimen to read as follows:


Parallel Section not less than 9"
Parallel within 0.010"
About 3"
$1\frac{1}{2}$" +1/8" −1/4"
8"±0.01" Gage Length for Measuring Elongation after Fracture
1" to 3" Radius
Approximately 2"

NOTE 1.—When necessary, it is permissible to use a narrower specimen, but in such a case the reduced portion shall be not less than 1 in. in width.

NOTE 2.—Punch marks for measuring elongation after fracture shall be made on the flat or on the edge of the specimen and within the parallel section; either a set of nine punch marks 1 in. apart, or one or more sets of 8-in punch marks may be used.

NOTE 3.—The dimension "t" is the thickness of the test specimen as provided for in the applicable material specifications.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

Section 18.—Renumber as Section 19 and change to read as follows by the addition of the italicized words and figures:

19. *For the test specimen illustrated in Fig. 1, the width should be measured at each end of the gage length to determine parallelism, and also at the center. The thickness should be measured at the center only and used with the center measurement of the width to determine the cross-section area. The center width dimension should be recorded to the nearest 0.005 in., and the thickness measurement to the nearest 0.001 in.* The dimensions of cross-section of *all other* tension test specimens shall be measured by means of a measuring instrument, preferably reading to 0.5 per cent of the dimensions measured and in any case to at least 0.001 in. The stress shall be calculated on the basis of the minimum cross-section as determined by measurement.

Section 19.—Renumber as Section 20 and change to read as follows:

20. *Speed of Testing.*—Speed of testing may be defined in terms of rate of relative movement of the heads of the testing machine, in terms of rate of stressing the specimen, or in terms of rate of straining the specimen. For many materials specifying the rate of relative movement of the heads is adequate; for some materials specifying rate of stressing may be preferable; for other materials specifying rate of straining is necessary in order to obtain test values within acceptable limits. Suitable limits for speed of testing should be specified for materials for which the differences resulting from the use of different speeds are of such magnitude that the test results are unsatisfactory for determining the acceptability of the material. In such instances, depending upon the material and the use for which it is intended, one or more of the methods described in the following Paragraphs (*a*) to (*c*) is recommended for specifying speed of testing:

(*a*) *Crosshead Speed.*—The allowable limits for the speed of the crosshead of the testing machine shall be specified in inches per minute. Unless otherwise indicated, crosshead speed shall mean the free-running or no-load speed of the crosshead of the testing machine. The limits for the crosshead speed may be further qualified by specifying different limits for various types and sizes of specimens. The average crosshead speed can be experimentally determined by using a suitable scale and stop watch.

(*b*) *Rate of Stressing.*—The allowable limits for rate of stressing shall be specified in pounds per square inch per minute. The average rate of stressing can be determined with a stop watch by observing the time required to apply a known increment of stress.

(*c*) *Rate of Straining.*—The allowable limits for rate of straining shall be specified in inches per inch per minute. The average rate of straining can be determined with a stop watch by observing the time required to effect a known increment of strain.

(*d*) Any convenient speed of testing may be used up to one-half the specified yield strength or yield point, or up to one-quarter the specified tensile strength, whichever is smaller. The speed above this point shall be within the limits specified. If different speed limitations are desired for use in determining yield strength, yield point, tensile strength, elongation, and reduction of area they should be stated in the product specifications.

NOTE.—In writing new standards, or in revising old standards, the A.S.T.M. product specifications committee will have the responsibility of deciding for any given material what method of measuring speed of testing is to be used, and of specifying suitable numerical limits for crosshead speed, rate of stressing, or rate of straining.

Standard Specifications for A.S.T.M. Thermometers (E 1 – 44):

The following revisions in this standard are recommended for immediate adoption:

Omit from the designations of each thermometer the symbol "E1" and also the parentheses.

All dimensions on both Fahrenheit and Centigrade thermometers to be in millimeters, except use inches for immersion designation on Fahrenheit thermometers.

Immersion line on thermometers to be approximately same length as longest graduation line.

Special markings on thermometers to be in capital letters without use of periods after abbreviations.

Add "Saybolt Vis." to requirements for special marking on A.S.T.M. Saybolt viscosity thermometers.

Substitute "PARAFFIN" for abbreviation "Pffe." on paraffin wax melting point thermometers 14C and 14F.

These recommendations have been

submitted to letter ballot of the committee which consists of 58 members; 44 members returned their ballots with the following results: E 8 – 42, 35 affirmative, 0 negative and 9 "not voting"; E 1 – 44, 31 affirmative, 0 negative and 13 "not voting."

ACTIVITIES OF TECHNICAL COMMITTEES

Technical Committee I on Mechanical Testing (H. F. Moore, chairman):

The activities of the sections of Technical Committee I have centered around problems which have arisen during the war period.

Section on Speed of Testing (P. G. McVetty, chairman).—The problems concerned with speed of testing have been difficult and important. They affect the work of other sections of Technical Committee I, especially the Section on Tension Testing and the Section on Elastic Strength. The Section on Speed of Testing held a meeting on February 26, 1946, in Pittsburgh, Pa. An extensive correspondence with critics of the present methods of specifying speed of testing now in the A.S.T.M. Standards has been carried on, and the section has, by letter ballot, voted to recommend a change in Section 19 of Standard Methods E 8 as given earlier in this report. The proposed revised Section 19 recognizes three methods of measuring speed of testing. (1) Measuring the rate of motion of the free running head of the testing machine, which seems satisfactory for tension tests of a number of materials, (2) Measuring the rate of application of stress, which seems satisfactory and feasible for some tests, and (3) Measuring the actual rate of straining, which is somewhat more difficult to use in routine tests, but seems to be necessary, or at least highly desirable for some materials.

In writing new standards, or in revising old standards the A.S.T.M. product specifications committee will have the responsibility for deciding for any given material what method of measuring speed of testing is to be used, and for specifying suitable numerical limits for crosshead speed, rate of stressing, or rate of straining.

Section on Tension Testing (R. L. Templin, chairman).—This section held a meeting on February 26, 1946, at Pittsburgh. The proposed requirements for speed of testing were discussed. Various difficulties in connection with shape and sizes of tension specimens were discussed, and arrangements made for cooperation with the Society committees on various materials, especially with Subcommittee XIII on Methods of Testing, of Committee A-1 on Steel. Consideration was given to problems arising in connection with tests of nonmetallic materials, and tension tests of powdered metals.

Section on Compression Testing (M. F. Sayre, chairman).—The revision of the Tentative Methods of Compression Testing of Metallic Materials (E 9 – 33 T) that has been under consideration by this section for the past several years is expected to be completed[1] in time for inclusion in the new edition of the Book of A.S.T.M. Standards.

Section on Elastic Strength (F. B. Seely, chairman).—The Standard Definitions of Terms Relating to Methods of Testing (E 6 – 36) are affected by the proposed change in Standard E 8, and consideration of these changes is planned at a meeting of the section to be held during the Annual Meeting.

Section on Calibration of Testing Machines and Apparatus (H. F. Moore, chairman).—This section held a meeting on February 27, 1946, at Pittsburgh. Three projects were discussed, and assigned to task groups for report at the June meeting. These projects were:

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[1] See Editorial Note, p. 475.

(*1*) The removal, as far as possible, of conflicts between the A.S.T.M. Standards for elastic calibration devices and the Bureau of Standards requirements for proving rings for testing machines. The task group for this work includes a representative from the Bureau of Standards, an extensive user of testing machines for routine tests, and an engineer from one of the large manufacturers of testing machines.

(*2*) A task group to study and prepare a draft of standard methods of calibrating large testing machines (machines of 1,000,000-lb. capacity and over). This task group consists of a representative of the Bureau of Standards, a representative of a large manufacturer of testing machines, and a member of the technical staff of one of the large U. S. Navy laboratories.

(*3*) The preparation of a draft which shall make the standards for the calibration of testing machines a separate standard from the standard for calibrating devices, especially elastic calibrating devices, such as proving rings, proving bars, etc.

Technical Committee II on Consistency, Plasticity, and Related Properties (Robert Burns, secretary):

This technical committee has suffered a severe loss in the death of its chairman, E. C. Bingham.

Due to the war effort it was necessary to defer active consideration of several committee projects. The general program of work will be reviewed at the meeting of the technical committee to be held during the Annual Meeting.

The Section on Methods and Apparatus for Absolute Viscosity Measurements (W. F. Fair, Jr.) plans to proceed with the compilation of available methods and apparatus used for determining absolute viscosity. A meeting of this section is also to be held during this annual meeting.

Technical Committee III on Particle Size and Shape (L. T. Work, chairman):

The Section on Pigment-Type Materials (C. E. Barnett, chairman) undertook early in the war a cooperative program of particle size measurements involving the measurement with the microscope of particles in mounted slides of pigment powders. No specifications on the optical equipment were given the cooperators so that a variety of apparatus and methods were used. The results obtained showed closer agreement than had been expected and are being studied by the section. The next step in this program will be to determine whether as good agreement can be obtained when the preparation of the microscopic mount is included in the variables studied. The entire program of this committee will be reviewed at a meeting to be held during this annual meeting.

Technical Committee IX on Interpretation and Presentation of Data (H. F. Dodge, chairman):

The increase in production resulting from the war effort placed added emphasis on standardization, and particularly on quality control. This resulted in expanding greatly the application of statistical methods. The A.S.T.M. Manual on Presentation of Data, which was prepared by this technical committee, was quite extensively used in the many studies undertaken in the improvement of the quality of products.

The interest in the subject of quality control has increased to such an extent that the Executive Committee decided to organize a new A.S.T.M. technical committee to promote the knowledge of quality control methods and their application to specifications and methods of test. This new committee will be designated Committee E-11 on Quality Control, and following its organization in

June it is planned to discontinue this E-1 technical committee.

Technical Committee X on Conditioning and Weathering (Robert Burns, chairman):

This technical committee has continued to assist other committees of the Society on problems relating to conditioning and accelerated weathering.

Consideration has been given to a revision of the Tentative Definitions with Procedures Relating to Conditioning and Weathering (E 41 – 42 T) as regards the requirements for "standard laboratory atmosphere," in order to establish such conditions for general use as "a relative humidity of 50 per cent at a temperature of 75 F. (21 C.)," unless otherwise specified. This revision is being discussed further with several Society committees.

It is recommended that the Tentative Recommended Practice for Characteristics of Standard Carbon Arc Accelerated Weathering Unit (E 42 – 42 T) be continued without change.

Technical Committee XI on Designation and Interpretation of Numerical Requirements (H. F. Dodge, secretary):

The committee records its appreciation of the excellent work of the former chairman of this technical committee, J. A. Gann, who found it necessary to resign due to a change in his business activities.

The activities of this technical committee are also to be transferred to the new Committee E-11 on Quality Control. It is accordingly planned to discontinue this technical committee when the new committee has been organized.

Technical Committee XII on Laboratory Apparatus (W. H. Fulweiler, chairman):

The sections of this technical committee have continued to cooperate and assist other standing committees on questions dealing with laboratory apparatus.

Section A on Thermometers (R. M. Wilhelm, chairman) has been especially active during the year and held two meetings in Philadelphia, Pa., on January 29, 1946, and April 22, 1946. At the request of Committee D-3 on Gaseous Fuels specifications for three new thermometers have been developed for use in the test for calorific value of fuels. These cover, respectively, a gas calorimeter inlet thermometer, outlet thermometer, and flue thermometer.

The section has recommended several changes in the Standard Specifications for A.S.T.M. Thermometers (E 1 – 44), as given earlier in this report.

Consideration is now being given by the section to specifications for a benzene freezing point thermometer, a Stormer viscosimeter thermometer, and additional kinematic viscosity thermometers for tests at 68, 70, 77, 122, 140 and 180 F.

At the request of Committee D-2, the section is also preparing specifications for a new thermometer for the congealing point test of pharmaceutical petrolatums, and a new thermometer for use in the test for oil content of paraffin wax.

These several new thermometer specifications are expected to be completed later this year and included in the Tentative Specifications for A.S.T.M. Thermometers (E 1 – 45 T).[1]

Section C on Volumetric Glassware (J. J. Moran, chairman) has been actively reviewing requirements for a number of pieces of volumetric glassware specified in methods recently issued by the Society. This has resulted in a number of improvements being effected. Several other recommendations are now under consideration by other technical committees, and it is expected that these will result in further changes being made later this year.

Technical Committee XIII on Hydrogen Ion Determinations (E. B. Ashcraft, chairman):

The Symposium on pH Measurement[2] being held at this annual meeting has been sponsored by this technical committee.

A Proposed Method for the Determination of the pH of Aqueous Solutions with the Glass Electrode, prepared by G. G. Manov at the request of the technical committee, is now being studied and will be recommended as tentative later in the year.[1]

The present officers of the committee have been re-elected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee, which consists of 58 members; 44 members returned their ballots, of whom 42 voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. H. FULWEILER,
Chairman.

P. J. SMITH,
Ex-officio Secretary.

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[2] Issued as separate publication, 1947.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method for:

The Determination of the pH of Aqueous Solutions with the Glass Electrode (E 70 - 46 T).

Revision of Tentative Specifications for:

ASTM Thermometers (E 1 - 46 T).

These recommendations were accepted by the Standards Committee on December 3, 1946, and the new and revised tentatives appear in the 1946 Book of A.S.T.M. Standards, Parts II, III-A, and III-B.

On December 5, 1946, the Administrative Committee on Standards accepted proposed revisions in the Tentative Methods of Compression Testing of Metallic Materials (E 9 - 33 T). The revised tentative methods appear in the 1946 Book of A.S.T.M. Standards, Parts I-A, and I-B, bearing the designation E 9 - 46 T.

REPORT OF COMMITTEE E-2*
ON
SPECTROGRAPHIC ANALYSIS

Committee E-2 on Spectrographic Analysis held a meeting in Buffalo, N. Y. on June 25 and 26, 1946, in connection with the Annual Meeting of the Society. At this meeting reports of progress were received from the chairmen of the various subcommittees, and action was taken on the reorganization of the committee as referred to later in this report.

On February 2, 1945, during the Group Committee Meetings in Pittsburgh, Pa., the officers of Committee E-2 met with the officers of Committee E-3 on Chemical Analysis of Metals, and C. L. Warwick and R. E. Hess of the Headquarters Staff, to discuss the overlapping scopes of Committees E-2 and E-3 over the fields of spectrophotometry and colorimetry. It was decided that even though, officially, these fields fall within the scope of Committee E-2, since Committee E-3 has developed methods utilizing them, the methods should remain under the jurisdiction of Committee E-3 provided Committee E-2 is kept informed about them. The scope of Committee E-2 was not changed. At some future date, Committee E-2 may wish to correlate visible, ultraviolet or infrared techniques used by other committees. It was also decided not to attempt to incorporate mass spectrometry in the scope of Committee E-2.

During the past year, there have been six additions in membership and one resignation, making the committee enrollment now 89.

A Symposium on Spectroscopic Light Sources[1] was arranged by a committee consisting of G. H. Dieke, J. Sherman, H. V. Churchill, M. E. Warga, and E. B. Ashcraft, chairman. The Symposium, comprised of the following four papers, was held at the Annual Meeting of the Society in Buffalo, N. Y. on June 25, 1946:

"The Present Status of Excitation in Spectrographic Analysis" by B. F. Scribner.

"A Study of the Controlled Spectrographic Spark Source," by J. H. Enns and R. A. Wolfe.

"Some Properties of Gas Discharges Used as Spectral Sources," By R. C. Mason, and

"Short Period Behavior of Spectroscopic Light Sources," by G. H. Dieke.

Committee E-2 is to be reorganized to consist of ten subcommittees as follows:

Subcommittee:

I on Apparatus and Equipment (K. B. Thomson, chairman),
II on Fundamental Methods and Techniques (E. B. Ashcraft, chairman),
III on Editorial (C. L. Geuttel, chairman),
IV on Standards and Pure Materials (C. H. Corliss, chairman),
V on Copper, Nickel, and Their Alloys (P. Leichtle, chairman),
VI on Lead, Tin, Antimony, Bismuth and Their Alloys (R. D. McLellan, chairman),
VII on Aluminum, Magnesium, and Their Alloys (J. R. Churchill, chairman),
VIII on Zinc, Cadmium, and Their Alloys (E. K. Jaycox, chairman),
IX on Ferrous Metals (P. Irish, chairman),
X on Nonmetals (R. H. Bell, chairman).

Mr. W. Boyer has been appointed liaison member with Committee E-3 on Chemical Analysis of Metals and C. Geuttel with Committee E-8 on Nomenclature and Definitions.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

[1] Issued as separate publication, March, 1947.

The main project of the committee for the coming year will be the writing and assembling of spectrochemical techniques to cover as many materials as possible that are of interest to members of Committee E-2.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, B. F. Scribner.
Vice-Chairman, E. B. Ashcraft.
Secretary, Mary E. Warga.

This report has been submitted to letter ballot of the committee, which consists of 89 members; 52 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. V. CHURCHILL,
Chairman.

MARY E. WARGA,
Secretary.

REPORT OF COMMITTEE E-3*

ON

CHEMICAL ANALYSIS OF METALS

Although only one meeting of Committee E-3 on Chemical Analysis of Metals has been held during the year—at Pittsburgh, Pa., on February 28, 1946—the members of the committee have been very active through meetings of the divisions and subcommittees. A serious effort has been made to prepare new methods and revise old ones where necessary in order that the 1946 edition of the Book of A.S.T.M. Methods of Chemical Analysis of Metals will be up to date. The members of Committee E-3 have contributed largely of their time for this work. The Advisory Committee met twice, first on November 5, 1945, at the National Bureau of Standards in Washington, D. C. and then on February 28, 1946, in Pittsburgh. Sixteen divisional and subcommittee meetings were held during the year as well as several informal meetings of groups to discuss certain specific phases of committee work.

Eighteen new members and one consulting member were added to the committee during the year. Two members transferred from active status to that of consulting membership. Two committee members resigned. The active membership of the committee is now 93.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, G. E. F. Lundell.
Vice-Chairmen, C. B. Francis.
H. A. Bright.
Secretary, J. W. Stillman.
Members-at-Large of Advisory Committee: T. R. Cunningham, Calvin Sterling, W. J. Boyer, B. F. Scribner.

At the direction of the Advisory Committee, a subcommittee studied the regulations of Committee E-3 and recommended certain revisions to bring them up to date and to improve the operations of the committee. The revised regulations were submitted to letter ballot and approved.

At Pittsburgh the chairman and secretary of Committee E-2 on Spectrographic Analysis met with one of the vice-chairmen and secretary of Committee E-3 together with Mr. Warwick and Mr. Hess of the A.S.T.M. staff and discussed the overlapping interest of the two committees with particular consideration of the scope of the two committees as printed in the A.S.T.M. Year Book. It was the consensus of opinion at this meeting that it would not be necessary to change the wording of the scope but that Committee E-3 would be free to develop and use photometric methods and other newer techniques as applied to the chemical analysis of metals.

Eighteen methods, either in the form of new procedures or revisions of present procedures, were submitted to letter ballot of Committee E-3. One procedure, the sampling of slab zinc, was rejected on the first ballot but later revised and approved by letter ballot. These various recommendations, which are listed in Table I, were presented to the Society through the Administrative Committee on Standards. Table I

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24, 1946.

TABLE I.—ANALYSIS OF LETTER BALLOT VOTE.

Items	Affirmative	Negative	Ballots Marked "Not Voting"
I. NEW TENTATIVES			
Chemical Analysis for Nitrogen in Steel (E 30 - 45 T), *accepted August 27, 1945;* supporting data in Table II	43	0	12
Chemical Analysis for Sulfur by Direct Combustion in Steel (E 30 - 46 T), *accepted April 26, 1946;* supporting data in Table III	48	1	2
Polarographic Determination of Lead and Cadmium in Zinc (E 68 - 46 T), *accepted April 26, 1946;* supporting data in Table IV	34	0	23
Chemical Analysis of Special Brasses and Bronzes (E 54 - 46 T), *accepted March 30, 1946;* supporting data in Table V	54	0	4
Sampling Wrought Non-Ferrous Metals and Alloys for Determination of Chemical Composition (E 55 - 46 T), *accepted March 30, 1946*	46	2	13
Chemical Analysis of White Metal Bearing Alloys (E 57 - 46 T), *accepted April 26, 1946;* supporting data in Tables VI and XIII	45	0	12
Photometric Determination of Bismuth in Pig Lead (E 58 - 45 T), *accepted August 27, 1945;* supporting data in Table VII	37	0	16
Recommended Practice for Photometric Methods for Chemical Analysis of Metals (E 60 - 46 T), *accepted March 30, 1946*	47	0	7
Photometric Methods for Chemical Analysis of Magnesium and Magnesium-Base Alloys (E 61 - 46 T), *accepted March 30, 1946;* supporting data in Table VIII	45	0	9
Photometric Method for Determination of Nickel in Copper and Copper-Base Alloys (E 62 - 46 T), *accepted January 10, 1946;* supporting data in Table IX	51	0	8
Photometric Methods for Chemical Analysis of Copper and Copper-Base Alloys, Determination of Phosphorus, Iron (Thiocyanate), Iron (Ferric Chloride) and Manganese (E 62 - 46 T), *accepted April 26, 1946;* supporting data in Table IX	41	1	15
Photometric Method for Determination of Iron in 70-30 Copper Nickel Alloy (E 63 - 46 T), *accepted March 30, 1946;* supporting data in Table X	47	0	7
Photometric Methods for Determination of Iron in Slab Zinc (Spelter) (E 64 - 46 T), *accepted March 30, 1946;* supporting data in Table XI	48	1	5
Sampling Slab Zinc (Spelter) (E 65 - 46 T), *accepted July 10, 1946*	46	2	13
Photometric Method for Determination of Iron in Lead- and Tin-Base Alloys (E 67 - 46 T), *accepted April 26, 1946;* supporting data in Table XII	43	0	14
II. REVISION OF TENTATIVES			
Recommended Practices for Apparatus and Reagents for Chemical Analysis of Metals (E 50 - 46 T), *accepted April 26, 1946*	48	0	9
Chemical Analysis of Copper (Electrolytic Determination of Copper) (E 53 - 46 T), *accepted January 10, 1946* (formerly Method B 34 - 36 T)	46	1	11
Chemical Analysis of Lead- and Tin-Base Solder Metal (E 46 - 46 T), *accepted July 10, 1946;* supporting data in Table XIII	47	0	14
III. REVISION OF STANDARD AND REVERSION TO TENTATIVE			
Chemical Analysis of Ferro-Alloys (E 31 - 46 T), *accepted March 30, 1946*	38	0	16

shows the results of the letter ballot vote in Committee E-3 and the date of acceptance by the Standards Committee. Supporting data obtained in cooperative tests using the procedures are given in Tables II to XIII. All of the new and revised methods will appear in the 1946 Book of A.S.T.M. Methods of Chemical Analysis of Metals.

ACTIVITIES OF DIVISIONS AND SUBCOMMITTEES

Division A on Ferrous Metals (Arba Thomas, chairman).—The Ferrous Division held one meeting during the year at Pittsburgh, but members were active on procedures for the analysis of iron, steel, and ferro-alloys. In the list of methods approved by the committee, three procedures were the result of work done in Division A.

A subcommittee has been at work on the procedure for the determination of tin in steel, and data collected so far indicate that the method is satisfactory. The method will be written up and submitted to subcommittee members for use in analyzing standard steel samples.

The subcommittee on aluminum in alloy steels is circulating a method for

TABLE II.—RESULTS OF COOPERATIVE TESTS ON THE TENTATIVE METHOD OF CHEMICAL ANALYSIS FOR NITROGEN IN STEEL (E 30 – 45 T).

Analyst*			Brown		Cunningham		Geyer	Boyer		Vance	Bright
Sample Number[a]	Type of Steel	Accepted Value for Nitrogen, per cent	Method E 30 – 45 T	Method E 30 – 45 T, 10-min. Digestion	Method E 30 – 45 T	Own Method	Method E 30 – 45 T	Method E 30 – 45 T	Own Method (HCl)	Method E 30 – 45 T	Semimicro Method (Allen)
13d	B.O.H., Carbon	...	0.0046	0.0042	....	....	0.0047	....	0.005	....	....
15c	B.O.H., Carbon	0.005[c]	....	....	0.0054	0.0076	....	....	....	....	0.004
125	5 per cent Si	0.005[d]	0.0060	0.0061	....	....	0.0066	0.007	0.007	....	....
563-1	Strip (4 per cent Si)	0.010[e]	0.0102	0.0075	....	....	....	....	...	....	....
50a	Cr-W-V	...	0.0202	0.0060	....	....	....	....	....	....	....
73a	13 Cr	0.036[c]	0.0355	0.0352	....	....	....	....	....	....	0.033
101a	18Cr-8Ni	0.044[c]	....	....	0.0497	0.0476	....	0.048	0.046	....	0.044
121	18Cr-8Ni-0.4Ti	0.009[e]	0.0085	0.0041	....	....	....	....	....	....	0.009
123	18Cr-8Ni-0.4Cb	...	0.0440	0.0437	0.045	0.043	0.0435	0.047	....	0.053	0.041
133	13Cr-0.6Mo	0.046[c]	0.0436	....	....	....	....	....	....	...	....
USS[b]	27Cr	...	0.234	0.232	0.222	0.225 0.221[f]	0.231	0.228	0.230	0.231[g] 0.232[h]	....

* The analysts were as follows: W. J. Brown, Nat. Lead Co.; T. R. Cunningham, Union Carbide & Carbon Research Labs., Inc.; C. O. Geyer, Inland Steel Co.; W. J. Boyer, Rustless Iron & Steel Div., The Am. Rolling Mill Co., E. R. Vance, Timken Roller Bearing Co., Steel & Tube Div.; H. A. Bright, Nat. Bureau Stds.

[a] These sample numbers all refer to National Bureau of Standards standard samples, unless otherwise indicated.

[b] United States Steel Corp. standard sample.

[c] National Bureau of Standards certificate value.

[d] Vacuum fusion, Arba Thomas, Armco Research Labs.

[e] Vacuum fusion, National Bureau of Standards.

[f] Dissolved in $HClO_4$, and residue Kjeldahled.

[g] 1-g. sample dissolved in 25 ml. of HCl (60 per cent).

[h] 1-g. sample dissolved in 20 ml. of $HClO_4$ (36 per cent) under reflux condenser.

TABLE III.—RESULTS OF COOPERATIVE TESTS ON THE TENTATIVE METHOD OF CHEMICAL ANALYSIS FOR SULFUR BY DIRECT COMBUSTION IN STEEL (E 30 – 46 T).

Sample Number[a]	Material	Accepted Value for Sulfur (Gravimetric Method), per cent	Analyst[b]		
			Steiner	Thomas	Boyer
5	Ingot iron	0.019	0.016	0.018	0.016
			0.017	0.018	0.017
			0.017	0.018	0.018
			0.018		
			0.018		
			0.019		
			0.020		
			Average...0.018	0.018	0.017
6	Carbon steel	0.272	0.264	0.273	0.265
			0.264	0.273	0.268
			0.268	0.276	0.268
			0.270		
			0.276		
			0.278		
			0.280		
			0.280		
			Average...0.273	0.274	0.267
7	Stainless steel, type 316	0.034	0.032	0.039	0.037
			0.035	0.039	0.037
			0.036	0.039	0.038
			0.038	0.041	
			0.038		
			Average...0.036	0.040	0.037
8	Stainless steel, type 303	0.312	0.284	0.308	0.313
			0.288	0.310	0.315
			0.288	0.313	0.318
			0.296		
			0.300		
			0.301		
			0.310		
			0.320		
			Average 0.298	0.310	0.315
9	Stainless steel, 18Cr-11Ni-0.4Ti	0.010	0.012	0.010	0.012
			0.013	0.011	0.012
				0.011	0.012
				0.012	
			Average...0.012	0.011	0.012

[a] Samples Nos. 5, 6, and 9 were National Bureau of Standards standard samples 55b, 129a, and 121a, respectively. Samples Nos. 7 and 8 were small lots of commercial steels, checked for gravimetric sulfur by members of Division A.

[b] The analysts were as follows: W. E. Steiner, Bethlehem Steel Co., Inc.; Arba Thomas, Armco Research Labs.; W. J. Boyer, Rustless Iron & Steel Div., The Am. Rolling Mill Co.

this determination with samples to subcommittee members for cooperative tests.

A method for the determination of boron has been prepared by the subcommittee in charge of this assignment and will be submitted to letter ballot of the division.

The subcommittee for the determination of bismuth in steel recommended that since there was very little demand for this method the subcommittee be relieved of the assignment, and this action was approved by the division.

TABLE IV.—RESULTS OF COOPERATIVE TESTS ON THE TENTATIVE METHOD FOR POLAROGRAPHIC DETERMINATION OF LEAD AND CADMIUM IN ZINC (E 68 - 46 T).

Sample	N.B.S. Certificate or Accepted Value		Laboratory No. 1		Laboratory No. 2		Laboratory No. 3	
	Lead, per cent	Cadmium, per cent	Lead, per cent	Cadmium, per cent	Lead, per cent	Cadmium, per cent	Lead, per cent	Cadmium, per cent
Sample A of special high-grade zinc	0.0027	0.0026	0.0024	0.0028	....	....	....	....
Sample B of special high-grade zinc	0.0004	0.0001	0.00044	0.0002	0.0006	0.0002	....	....
N.B.S. sample 108	0.047	0.092	0.051	0.103	0.045	0.094	0.0505	0.096
N.B.S. sample 109	0.0020	0.0018	0.0023	0.0023	0.0018	0.0017	0.0025	0.0019

COMPARISON OF RESULTS FROM DETERMINATION OF LEAD IN ZINC BY THE POLAROGRAPHIC METHOD (E 68 - 46 T) AND BY THE ELECTROLYTIC METHOD

Sample Number	Lead, per cent	
	Electrolytic Method (100-g. sample)	Polarographic Method (1-g. sample)
4B	0.0038	0.0036
2C	0.0039	0.0040
9C	0.0038	0.0036

The subcommittee on the determination of beryllium has submitted methods for trial by subcommittee members and is making progress toward a satisfactory procedure.

In addition to the specific assignments mentioned, the Ferrous Division has been critically examining the present methods for the analysis of iron, steel, and ferro-alloys with a view to improving the procedures in the light of recent developments in the field.

Division B on Non-Ferrous Metals (D. R. Evans, chairman).—The Non-Ferrous Division has held two meetings during the year, one at the Bell Telephone Laboratories at Murray Hill, N. J., and New York, N. Y., in the fall and the other at Pittsburgh in the spring. Very active attention has been given to the development and revision of methods for publication in the 1946 edition of the Book of Chemical Analysis of Metals. Twelve of the methods in the list approved by the committee were sponsored by Division B. Advantage has been taken of the newly developed instruments. As an example, two of the methods issued use the polarograph. It is expected that this development will lead to the formation of a new subcommittee to investigate further applications of this technique. Subcommittee B-7 on Photometric Methods has been especially active in developing new methods in this field for application to non-ferrous alloys.

Division B is making plans to continue the development of methods for the constituent elements of non-ferrous alloys using, where applicable, the newer techniques of photometric and polarographic procedures. Among the activities of subcommittees which are already under way are the following:

1. A study is being made of methods for the analysis of lead.

2. The photometric method for the determination of aluminum, which was rejected on letter ballot, is being given further study.

3. Photometric methods for use in the analysis of aluminum alloys are being investigated.

The subcommittee on nickel has under consideration methods for the analysis of nickel for electronic applications and also for the analysis of monel metal. An alloy of nickel-silver has been prepared

TABLE V.—RESULTS OF COOPERATIVE TESTS ON THE TENTATIVE METHODS OF CHEMICAL ANALYSIS OF SPECIAL BRASSES AND BRONZES (E 54 - 46 T).

Sample	Laboratory No. 1	Laboratory No. 2	Laboratory No. 3
Arsenic by the Distillation - Iodimetric Method, per cent			
Leaded bronze[a]	1.23 1.22 1.26 1.22 1.21 1.20 1.23 1.20 1.20[b] 1.21[b]	1.19 1.21 1.18	1.18 1.16
Phosphor bronze, N.B.S. sample 63a (Certificate value, 0.027 per cent As)	0.027 0.028		
Antimony by the Distillation - Iodimetric Method, per cent			
Leaded bronze[a]	1.00 1.00 1.01 1.00 1.02 0.97[b] 0.98[b]	0.99 0.97 1.01 0.96[b] 0.99[b]	1.00 1.02
Phosphor bronze, N.B.S. sample 63a (Certificate value, 0.49 per cent Sb)	0.47 0.47		
Phosphor bronze, N.B.S. sample 124 (Certificate value, 0.23 per cent Sb)	0.24		
Tin by the Iodimetric Titration Method, per cent			
Leaded bronze[a]	9.99 10.00 10.00 9.98	9.99 10.04 9.98 10.02	9.98

[a] The nominal composition of the leaded bronze sample was as follows:

Copper, per cent	81
Tin, per cent	10
Arsenic, per cent	1
Antimony, per cent	1
Lead, per cent	5
Phosphorus, per cent	0.75
Zinc, per cent	1

[b] These values were determined by a different method.

TABLE VI.—RESULTS OF COOPERATIVE TEST ON THE TENTATIVE METHODS OF CHEMICAL ANALYSIS OF WHITE METAL BEARING ALLOY (E 57 - 46 T).

Laboratory	A	B	C	D	E	F
Copper, per cent	3.68[a] 3.70[a] 3.70[a]	3.69 3.68 3.70 3.72 3.71 3.70	3.67 3.66 3.72 3.70 3.72 3.66 3.69[a] 3.69[a] 3.68[a] 3.70[a]	3.65 3.68 3.71 3.72 3.71 3.69	3.66 3.66	
Lead, per cent	5.05 5.03 4.99	5.03 5.03 5.01 4.99 5.01 4.97		4.96[b] 4.95[b] 4.94[b] 5.10	5.15[b] 5.13[b]	
Tin, per cent		82.70 82.70 82.62 82.71 82.66 82.69 82.66 82.66		82.79 82.58		82.7
Antimony, per cent	8.27 8.28 8.28	8.28 8.28 8.34		8.33 8.31 8.32		
Arsenic, per cent				0.106 0.114 0.105 0.109 0.111		0.11 0.10
Iron, per cent		0.010 0.011		0.012 0.012		

[a] Determined by the rapid electrolytic method (Sections 16 and 17 of Methods E 57).

[b] Determined by the sulfate method (Section 15 to (e) of Methods E 57).

TABLE VII.—RESULTS OF COOPERATIVE TEST ON THE TENTATIVE PHOTOMETRIC METHOD FOR DETERMINATION OF BISMUTH IN PIG LEAD (E 58 - 45 T).

Sample Number	Bismuth, per cent			
	Laboratory No. 1	Laboratory No. 2	Laboratory No. 3	Spectrograph
3	0.0034 0.0034 0.0034 0.0033	0.0028 0.0028	0.0030 0.0030	0.0032
8	0.025 0.025 0.025 0.025 0.025	0.026 0.026	0.026 0.0265	0.027
11	0.102 0.102 0.102 0.102 0.102	0.100 0.100	0.102 0.103	0.10

by the National Bureau of Standards, and a subcommittee is collaborating on its analysis.

Division C on Sampling (W. C. Bowden, Jr., chairman).—The Sampling Division has been reorganized during the year under the new chairman. Two meetings of the division have been held, one at Newark, N. J. and the other at Pittsburgh. Two of the methods voted upon by the committee were sponsored by Division C. In organizing the division, subcommittees will be set up only to carry out specific assigned tasks.

TABLE VIII.—RESULTS OF COOPERATIVE TESTS ON THE TENTATIVE PHOTOMETRIC METHODS FOR CHEMICAL ANALYSIS OF MAGNESIUM AND MAGNESIUM-BASE ALLOYS (E 61 - 46 T).

Element	Percentages Found by Methods E 61	Averages from A.S.T.M. 1942 Cooperative Tests	Number of Determinations	Type of Method
		A.S.T.M. Alloy 16035		
Fe	0.031 (Bipyridine) 0.031 (Bipyridine)	0.0335 0.0357	8 7	colorimetric volumetric
	0.031 (HBr) 0.027 (HBr)			
Cu	0.055 0.060	0.057	7	volumetric (iodide)
Mn	0.407 0.418	0.414 0.392	6 8	bismuthate persulfate
Si	0.184	0.180 0.184	6 3	H_2SO_4 dehydration $HClO_4$ dehydration
Pb	0.0023 0.0024	not determined		
		A.S.T.M. Alloy 16036		
Fe	0.0318 (Bipyridine) 0.0320 (Bipyridine)	0.0342 0.0338	8 8	colorimetric volumetric
	0.030 (HBr) 0.027 (HBr)			
Cu	0.047 0.049	0.0494 0.051	8 5	electrolytic volumetric (iodide)
Mn	0.340 0.327	0.321 0.321	6 8	bismuthate persulfate
		Apex Alloy A2005 A2		
Pb	0.033	not determined	1 1	spectrographic (Dow), 0.036 per cent spectrographic (Apex), 0.03 per cent

The question of what constitutes an adequate sample for slab zinc, when consideration is given to the possible burden caused by sampling too many slabs, was considered by the division. The first sampling procedure submitted to vote was rejected on this latter ground, but a revised procedure was later approved.

Division D on General Analytical Methods (S. E. Q. Ashley, chairman).—Division D has also been reorganized during the year. Two meetings of the division have been held, one at Pittsburgh and the other at Princeton, N. J. Four subcommittees have been set up as follows:

- D-1 on Reagents, W. M. Murray, Jr., chairman,
- D-2 on Apparatus, A. H. Bushey, chairman,
- D-3 on Common Procedures, and
- D-4 on Precision and Accuracy of Methods, Grant Wernimont, chairman.

The Tentative Recommended Practices for Reagents and Apparatus (E 50 -

TABLE IX.—RESULTS OF COOPERATIVE TESTS ON THE TENTATIVE PHOTOMETRIC METHODS FOR CHEMICAL ANALYSIS OF COPPER AND COPPER-BASE ALLOYS (E 62 - 46 T).

NICKEL BY THE DIMETHYLGLYOXIME METHOD, PER CENT

Analyst	Nickel Found	Standard Used for Calibration Factor[a]
1	0.534, 0.538, 0.534, 0.534	...
2	0.528, 0.526, 0.526, 0.523, 0.526	37c
3	0.524 to 0.545	standard nickel solution
4	0.53, 0.53	52b, 37b
5	0.53, 0.53	124, 62a
6	0.545 (average of 7 tests)	37c, 124, 62a, 62b
7	0.528, 0.526, 0.534, 0.532, 0.528	37c
8	0.537 to 0.545	62b

PHOSPHORUS BY THE MOLYBDIVANADOPHOSPHORIC ACID METHOD, PER CENT

(Deoxidized Copper and Phosphorized Brasses)

Sample	Laboratory No. 1	Laboratory No. 2	Laboratory No. 3
U.S.M.R. sample No. 556430	0.0228 0.0228 0.0227	0.0225 0.0226 0.0223	0.0227 0.0227

PHOSPHORUS BY THE MOLYBDIVANADOPHOSPHORIC ACID METHOD, PER CENT

(Copper-Base Alloys Containing 0.01 to 1.2 per cent Phosphorus)

Sample	Analyst No. 1	Analyst No. 2	Analyst No. 3	Analyst No. 4	Analyst No. 5
N.B.S. sample 63a (Certificate value, 0.58 per cent P)	0.586 0.582 0.580 0.580	0.577 0.567 0.576	0.60 0.59 0.60 0.59	0.580 0.575 0.560	0.58
N.B.S. sample 124 (Certificate value, 0.037 per cent P)	0.0372 0.0371 0.0371			0.0384	
Special sample (Established value, 0.194 per cent P)		0.194 0.200 0.194 0.193			

IRON BY THE THIOCYANATE METHOD, PER CENT

Sample	Analyst No. 1	Analyst No. 2	Analyst No. 3	Analyst No. 4
N.B.S. sample 37d	0.079 0.078	0.074 0.075	0.076 0.076 0.073	0.077
N.B.S. sample 62b (Certificate value, 0.82 per cent Fe)	0.82 0.83	0.82 0.84	0.80 0.83 0.80 0.80 0.81 0.84 0.84	0.83 0.83 0.83 0.84 0.83
N.B.S. sample 63a (Certificate value, 0.52 per cent Fe)	0.51 0.51 0.52	0.51 0.53	0.52 0.52 0.51	
N.B.S. sample 62a (Certificate value, 1.04 per cent Fe)	1.05 1.01 1.02	1.03 1.06		1.04 1.03 1.03 1.04 1.03

IRON BY THE FERRIC CHLORIDE METHOD, PER CENT

Sample	N.B.S. Certificate Value for Iron	Iron Found			
		Laboratory No. 1	Laboratory No. 2	Laboratory No. 3	Laboratory No. 4
Phosphor bronze, N.B.S. sample *63a*	0.52	0.52 0.53 0.53 0.53	0.51 0.50	0.53 0.52	
Sheet brass, N.B.S. sample 37b	0.21	0.18 0.19	0.18 0.19		
Sheet brass, N.B.S. sample 37c	0.17	0.17 0.17			0.17 0.17 0.17
Cast bronze, N.B.S. sample 52b	0.032	0.028 0.030 0.028		0.027 0.027	
Ounce metal, N.B.S. sample 124	0.38	0.37 0.37		0.38 0.37	0.38 0.38

MANGANESE BY THE PERIODATE METHOD, PER CENT

Sample	N.B.S. Certificate Value for Manganese	Laboratory No. 1	Laboratory No. 2	Laboratory No. 3[b]	
				Method A	Method B
Manganese bronze, N.B.S. sample 62	1.59			1.61 1.61 1.59 1.62 1.62	1.59 1.60 1.59 1.59
Manganese bronze, N.B.S. sample 62a	1.50	1.53 1.50	1.51 1.50 1.51 1.52		
Manganese bronze, N.B.S. sample 62b	1.29	1.30 1.30	1.27 1.29 1.28 1.27	1.29 1.31 1.28 1.28	1.30 1.30 1.28 1.31
"A.S.T.M." silicon bronze	0.99	1.00 0.99	0.99 0.99 1.00 1.00		

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[a] These sample numbers refer to National Bureau of Standards standard samples except in the case of the standard nickel solution.

[b] Method A used by Laboratory No. 3 was essentially the same as the periodate method described in Sections 42 to 48 of A.S.T.M. Methods E 62, which was used by Laboratories Nos. 1 and 2. In Method B, a 1.000-g. sample was treated as in Section 47 (*b*) and (*d*) and diluted to 100 ml., a 5-ml. aliquot was taken, and 10 ml. of HNO_3 and 5 ml. of H_3PO_4 added. The analysis was then continued in accordance with Section 47 (*f*) and (*g*).

TABLE X.—RESULTS OF COOPERATIVE TESTS ON THE TENTATIVE PHOTOMETRIC METHOD FOR DETERMINATION OF IRON IN 70-30 COPPER-NICKEL ALLOY (E 63 - 46 T).

Sample	Iron Content, per cent		
	Laboratory No. 1	Laboratory No. 2	Laboratory No. 3
70-30 copper-nickel alloy....	0.426	0.428	0.426
	0.426	0.428	0.430
	0.429	0.428	
	0.429	0.426	
	0.426		
	0.426		
	0.428		
	0.430		

[a] Volumetric determination of iron, in the same sample, with ceric ammonium sulfate gave the value of 0.43 per cent iron for each of three determinations.

TABLE XI.—RESULTS OF COOPERATIVE TESTS ON THE TENTATIVE PHOTOMETRIC METHODS FOR DETERMINATION OF IRON IN SLAB ZINC (SPELTER) (E 64 - 46 T).

Sample	Iron Content, per cent		
	Laboratory No. 1	Laboratory No. 2	Laboratory No. 3
	Salicylate Method	Thiocyanate Method	Ferric Chloride Method
Cooperative sample	0.0213	0.0213	0.020
	0.0213	0.0210	0.019
	0.0212	0.0212	
	0.0210	0.0212	
		0.0210	
		0.0214	
		0.0212	
N.B.S. sample 108 (Certificate value, 0.031 per cent Fe)...	0.0310		
	0.0313		
	0.0314		
	0.0310		
	0.0313		
	0.0311		
N.B.S. sample 110 (Certificate value, 0.014 per cent Fe)...	0.0142		
	0.0142		

46 T) submitted to letter ballot was revised by Division D.

Under the auspices of Division D, a Symposium on Polarographic Analysis was held at Princeton with considerable success. The attendance was limited to the members of the committee and invited guests.

TABLE XII.—RESULTS OF COOPERATIVE TESTS ON THE TENTATIVE PHOTOMETRIC METHOD FOR DETERMINATION OF IRON IN LEAD- AND TIN-BASE ALLOYS (E 67 - 46 T).

Sample	N.B.S. Certificate Value for Iron, per cent	Iron Found, per cent			
		Laboratory No. 1	Laboratory No. 2	Laboratory No. 3	Laboratory No. 4
A.S.T.M. white metal bearing alloy.......		0.012	0.011	0.011	
		0.013	0.010	0.012	
Tin-base, N.B.S. sample 54*b*............	0.028	0.028	0.023	0.027	0.027
		0.026	0.024	0.027	0.027
		0.025			
		0.026			
Lead-base, N.B.S. sample 53*b*..........	0.002	none	0.001		
		none	0.002		
Solder, N.B.S. sample 127..............		0.001	0.001		
		0.001	0.001	<0.001	

TABLE XIII.—RESULTS OF COOPERATIVE TESTS ON THE POLAROGRAPHIC METHOD FOR DETERMINATION OF ZINC IN LEAD- AND TIN-BASE ALLOYS (E 46 - 46 T AND E 57 - 46 T).

Sample	Zinc Content, per cent	
	Laboratory D	Laboratory G
N.B.S. sample No. 127	0.0012	0.0012
	0.0019	0.0013
	0.0013	0.0019
	0.0019	

Topics suggested for future symposia are oxidation-reduction procedures and precision and accuracy in the analysis of metals.

This report has been submitted to letter ballot of the committee, which consists of 93 members; 63 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

G. E. F. LUNDELL,
Chairman.

J. W. STILLMAN,
Secretary.

REPORT OF COMMITTEE E-4* ON METALLOGRAPHY

Committee E-4 on Metallography has not met since its last previous meeting on June 29, 1944. However, most of its subcommittees and the Advisory Committee held meetings during the A.S.T.M. Spring Group Committee Meetings at Pittsburgh, Pa., during the week of February 25, 1946.

The gains and losses in E-4 membership during the past two years have balanced out, with the total membership remaining at 73.

In common with the Society and many of its committees, Committee E-4 deeply regrets the passing away of C. H. Davis, one of our best officers, hardest workers, and a staunch friend.

ADOPTION OF TENTATIVE AS STANDARD

The committee recommends the adoption as standard of the Tentative Classification of Austenite Grain Size in Steels (E 19 - 39 T) and accordingly asks that this recommendation be approved at the Annual Meeting for reference to letter ballot of the Society.

This recommendation has been referred to letter ballot of the committee, which consists of 73 members; 43 members returned their ballots of whom 37 have voted affirmatively, 0 negatively, and 6 members marked their ballots "not voting."

REVISION OF TENTATIVES

The committee recommends that the Tentative Recommended Practice for Determining the Inclusion Content of Steel (E 45 - 42 T) be revised as follows and continued as tentative:

Section 1.—Add a second sentence to read as follows: "This practice deals only with recommended test methods and nothing in it should be construed as defining or establishing limits of acceptability for any grade of steel."

In the note, section entitled "Microscopic Methods," in the first paragraph, start a new paragraph at the sentence beginning "Various reference charts of this nature . . . ". Insert the following after this sentence: "In the JK chart the thin and heavy series of inclusions are shown to scale, and the nominal thicknesses of the inclusions are also recorded above each column. These values for the thicknesses of inclusions are not intended to be exact but only to permit approximate classifications."

Start a new paragraph at the sentence beginning "No chart can represent all . . .".

Figure 5.—At the top of the chart, under all columns entitled "Heavy Series" delete the word "up to" in the inscriptions "Thicknesses up to . . .".

At the bottom of the chart, after the line under the title, insert the following: "The numerical values in microns at the top of this chart are approximate; the classification of the inclusions into thin and heavy should be based only on comparison with the illustrations shown in the chart."

Section 2 (c).—Delete the sentence "The procedures discussed here apply only to bars or billets about 1 to 5 in. in diameter."

Section 5(c).—Delete the words "such as carbon tetrachloride."

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

Section 6(c).—In the second sentence, substitute the words "a quick drying solvent" for the words "carbon tetrachloride."

Section 7(b).—Add the following: "Indications can also be recorded by dipping into a suitable transparent plastic. After the plastic coating has been dried it can be removed and mounted."

Section 8.—Change the table in Paragraph (*a*) to read as follows:

Length of Inclusion, in.	Weight
1/16 to 1/4	1
Over 1/4 to 1/2	2
Over 1/2 to 3/4	4
Over 3/4 to 1	8
Over 1	16

Change the note in Paragraph (*f*) to read as follows, deleting the present reference to footnote 4: "Note.—Another method for rating inclusions by the magnetic powder method has been adopted by the Society of Automotive Engineers as S.A.E. Recommended Practice for Magnetic Particle Testing, 1945 S.A.E. Handbook, p. 327."

Section 11 (b).—At the end of the last paragraph add the following as a new paragraph: "If desired, the predominant type of inclusions may be recorded, whether they are sulfides, silicates, or oxides."

The committee recommends that the Tentative Recommended Practice for Identification of Crystalline Materials by the Hanawalt X-Ray Diffraction Method (E 43 – 42 T) be revised as follows and continued as tentative:

Section 8.—Reletter the present Paragraph (*b*) as (*c*) and add the following as a new Paragraph (*b*):

(*b*) *Calculating Interplanar Spacings.*—The measurements described in Paragraph (*a*) are not used directly in the index for the identification of crystalline materials, because the distances between corresponding lines depend upon the radius of the camera and the type of radiation used as well as upon the crystal structure of the sample. The patterns are described uniquely in terms of the interplanar spacings $\left(\frac{d}{n}\right)$ corresponding to the diffraction lines.[a] These interplanar spacings are calculated from the measured positions of the lines by using Bragg's equation:

$$\frac{d}{n} = \frac{\lambda}{2 \sin \theta}$$

where:

$\frac{d}{n}$ = the first order spacing,

λ = wave length of the X-rays, and

θ = diffraction angle. For the cylindrical camera recommended above, θ (in degrees) is calculated using the following equation: $\theta = \frac{45l}{\pi r}$ where l is the distance between corresponding lines on opposite sides of the undeviated beam, and r is the radius of the film. Many cameras are made with a radius of 57.3 mm. so that 1 mm. on the film equals 1 degree of arc. The best way to measure r is to make a pattern of a material with an accurately known structure (for example, NaCl) and to calculate r from some of the lines of that pattern. When a laboratory makes all powder patterns with the same type of camera and the same type of X-rays, it is well worth the effort to make a scale which reads the interplanar spacings directly from the film.

These recommendations have been referred to letter ballot of the committee which consists of 73 members; 39 members returned their ballots, with the following results: E 45, 32 affirmative, 0 negative, and 7 members marked their ballots "not voting"; E 43, 29 affirmative, 0 negative, and 9 members marked their ballots "not voting."

Activities of Subcommittees

Subcommittee I on Selection and Preparation of Samples (G. F. Comstock-chairman).—This group has revised the present Tentative Methods of Preparation of Metallographic Specimens (E 3 – 44 T), bringing them thoroughly up-to-date in accordance with best modern practice.

In order to facilitate the work of this subcommittee, and to give more adequate coverage in the field of macroetching, a separate subgroup has been authorized, and should be in effective operation during the ensuing year.

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[a] All data which are submitted to the committee for inclusion in the index should be in terms of $\frac{d}{n}$.

Subcommittee II on Definitions.—This subcommittee was discharged several years ago, but present demands indicate the advisability of resuming this activity. This is now in progress. The Standard Definitions of Terms Relating to Metallography (E 7 – 27), which had been under the jurisdiction of the former subcommittee, are recommended for retention for the time being.

Subcommittee IV on Photography (L. V. Foster, chairman).—This subcommittee has reviewed the Tentative Methods of Preparation of Micrographs of Metals and Alloys (Including Recommended Practice for Photography as Applied to Metallography) (E 2 – 44 T) under its jurisdiction, and recommended that they be continued as tentative, without change.

In addition to the above, this subcommittee has been active in sponsoring a Section on Photomicrography in the Photographic Exhibit at this Annual Meeting. Special commendation should be given to Messrs. L. V. Foster and R. F. Cameron for this excellent work.

Subcommittee VI on X-Ray Methods (W. L. Fink, chairman).—This subcommittee has reviewed the Tentative Recommended Practice for Identification of Crystalline Materials by the Hanawalt X-Ray Diffraction Method (E 43 – 42 T), and has recommended its retention as tentative, with but the addition of an explanatory paragraph, as given earlier in this report.

This subcommittee cooperated with the Joint Committee on Chemical Analysis by X-Ray Diffraction Methods in making a survey of present methods. It is anticipated that an acceptable method can be established.

Subcommittee VII on Methods of Thermal Analysis (L. H. Carr, chairman).—This subcommittee has not been active during the year. The Standard Recommended Practice for Thermal Analysis of Steel (E 14 – 33) is recommended for continuation without change.

Subcommittee VIII on Grain-Size (M. A. Grossman, chairman).—This subcommittee has recommended the adoption as standard of the Tentative Classification of Austenite Grain Size in Steels (E 19 – 39 T).

Subcommittee IX on Inclusions (Samuel Epstein, chairman).—This group has met twice during the year and has completed the several changes, listed earlier in this report, primarily clarifications of meaning, in the present Tentative Recommended Practice for Determining the Inclusion Content of Steel (E 45 – 42 T).

The Nominating Committee has submitted the following nominations for officers of the committee for the ensuing term of two years:

For Chairman, L. L. Wyman.
For Vice-Chairman, R. E. Penrod.
For Secretary, Mary Norton.

The election of officers was held during the June meeting of the committee with the above nominees being unanimously elected.

With the termination of the June meeting, Committee E-4 is losing two of its oldest and most active members, both of whom are retiring from office at their own request. Mr. J. J. Bowman, Secretary, and Mr. G. F. Comstock, Chairman of Subcommittee I, have indeed earned the thanks and gratitude of the committee. Mr. J. R. Vedilla will replace Mr. Comstock as chairman of Subcommittee I.

This report has been submitted to letter ballot of the committee, which consists of 73 members; 35 members have returned their ballots, of whom 34 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. L. WYMAN,
Chairman.

J. J. BOWMAN,
Secretary.

REPORT OF COMMITTEE E-6*

ON

METHODS OF TESTING BUILDING CONSTRUCTIONS

During the past few years the introduction of new materials, new methods of construction, and improved techniques in methods of assembly in the building field has created a strong demand for standard methods of testing to determine the acceptability of these advances in the building art. The need for such standards has been especially felt by building officials and prefabricators who of necessity have been compelled to improvise tests to meet their immediate problems but who at the same time have been keenly aware of the advantages of uniform nationally accepted methods of testing.

Recognition of the need for expansion of the activities of the Society in the field of testing parts and assemblies led to a recommendation by the Society's Administrative Committee on Simulated Service Testing that an A.S.T.M. technical committee on methods of testing building constructions be established. A special organizing committee under the chairmanship of D. E. Parsons was appointed to explore this project further. The committee met in Washington, D. C., on September 24, 1945, and after thorough discussion of the objectives of the program, drafted an appropriate scope and recommended personnel to serve on the proposed committee. The formation of the new committee, designated committee E-6 on Methods of Testing Building Constructions, on the Recommendation of the Administrative Committee on Simulated Service Testing, was approved by the Executive Committee of the Society on October 16, 1945, with the following scope:

> *Scope.*—To formulate methods of test for building constructions, including elements, connections, and assemblies, under actual or simulated service conditions, applicable to the evaluation of such factors as materials, design, construction, and fabrication.

Committee E-6 was organized at a meeting held in Washington, D. C. on January 23, 1946, with the following personnel:

Aluminum Company of America, E. C. Hartmann,
American Institute of Architects, Department of Technical Services, Theodore I. Coe,
American Iron and Steel Inst., B. L. Wood,
American Standards Assn., J. H. Courtney,
Associated General Contractors of America, B. L. Knowles,
Building Officials Conference of America, Inc., The, W. D. Guion,
Douglas Fir Plywood Assn., N. S. Perkins,
Federal Public Housing Authority, A. M. Korsmo,
J. M. Frankland, Chance Vought Aircraft Div. of United Aircraft Corp.,
Gypsum Assn., H. J. Schweim,
T. R. Higgins, Am. Inst. of Steel Constr.,
National Lumber Manufacturers Assn., F. J. Hanrahan,
L. J. Markwardt, U. S. Forest Products Lab.,
Owens-Corning Fiberglas Corp., T. S. Rogers,
D. E. Parsons, Nat. Bureau of Standards,
Pittsburgh Testing Laboratory, A. R. Ellis,
Portland Cement Assn., A. J. Boase,
Public Buildings Administration, Federal Works Agency, Peter Hein,
Purdue Research Foundation, C. F. Boester,
G. M. Rapp, John B. Pierce Foundation,
F. E. Richart, Univ. of Ill.,
Structural Clay Products Inst., H. C. Plummer,

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

Underwriters' Laboratories, Inc., Hugh M. Robinson,
University of California, Roy W. Carlson,
U. S. Department of Agriculture, Division of Farm Structures, W. J. Ashby,
U. S. Navy, Bureau of Yards and Docks, Code F-4-4, H. A. Stacy,
W. C. Voss, Massachusetts Inst. of Tech.,
K. F. Wendt, Univ. of Wis., and
H. L. Whittemore, Nat. Bureau of Standards.

At the organization meeting, committee officers were elected, the scope of the project was approved, and a tentative subcommittee structure was outlined. The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, L. J. Markwardt.
Vice-Chairman, F. E. Richart.
Secretary, J. H. Courtney.

The following subcommittees have since been organized:

Subcommittee I on Panels for Light-Building Construction, H. L. Whittemore, chairman.

Subcommittee II on Connections and Assembled Structures, G. M. Rapp, chairman.

Subcommittee III on Large Structural Units (trusses, girders, arches), F. E. Richart, chairman.

Subcommittee IV on General Structural Elements (joists, posts and columns, and elements not covered by Subcommittee I), K. F. Wendt, chairman.

An advisory committee has been appointed to consider the field of work for the committee and to act on special tests. The advisory committee will also serve as a standing committee to which industry can bring its problems.

The present subcommittee structure is not regarded as fixed in any sense but rather carries out the committee's concept of its function, namely, that of a task force which would attack first the most immediate and pressing problems, such as, for example, those in the housing field, expanding its organization as the need becomes apparent. First consideration is being given to methods of conducting strength tests of various kinds, because some such methods have already been employed and will serve as a basis for committee consideration. As the work progresses, attention will be given to other phases of the problem and to methods for some of the more intangible but nonetheless important requirements, such as durability and weathering tests.

This report has been submitted to letter ballot of the committee which consists of 32 members; 30 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

L. J. MARKWARDT,
Chairman.

J. H. COURTNEY,
Secretary.

TECHNICAL PAPERS

With Discussions

EDGAR MARBURG LECTURE

The purpose of the Edgar Marburg Lecture is to have described at the Annual Meetings of the American Society for Testing Materials, by leaders in their respective fields, outstanding developments in the promotion of knowledge of engineering materials. Established as a means of emphasizing the importance of promoting knowledge of materials, the Lecture honors and perpetuates the memory of Edgar Marburg, first Secretary of the Society.

PROTECTIVE ORGANIC COATINGS AS ENGINEERING MATERIALS[1]

TWENTIETH EDGAR MARBURG LECTURE

BY JOSEPH J. MATTIELLO[2]

INTRODUCTION

The manufacture of protective organic coatings is finally emerging as an industrial science. The term "Organic Coatings," as it is used in this lecture, includes such materials as varnishes, paints, enamels, lacquers (clear and pigmented), textile coatings, plastics, and similar special coating materials. It should be pointed out, however, that the growth of these coating materials has been primarily a development of the arts. A decade ago the industry was in process of transition; it had been on the threshold of becoming an industrial science for at least several decades. But, owing to the impetus of war work, the chemists, chemical engineers, and other technically trained men of the industry have not until the present had an opportunity to emphasize that the protective organic coating industry is a chemical and engineering industry.

These coatings are used for one or more of four main purposes: for decoration, for the protection of structural and technical materials, for sanitation, and for obtaining better distribution of light or greater illumination of objects. These functions of protective organic coatings were discovered or developed in the order named. For many centuries coating materials were used largely for their aesthetic appeal. Later as they were introduced into northern countries where works of art did not withstand exposure to the more severe climate, the protective aspects became of greater importance. Their use for sanitation and illumination are a result of the influence of science and of the modern mechanical age.

In the United States, Watin, in 1773, was the first to describe the paint and varnish industry as we know it today, technically. Copals and amber were the principal varnish resins at the time of the American Revolution. The resins were sweated before incorporation, and the oil was sweated to purify it. The thinner was turpentine.

The first varnish factory was established in England in 1790, in France in 1820, in Germany in 1830, and in Austria in 1843. Great Britain and the Netherlands were the first to place varnish manufacture upon a truly technical basis.

CHEMISTS' PART IN WAR WORK

The technical men of the protective coating industry directly concerned with the work of the recent World War had three acute problems: shifting availability of raw materials, conservation of raw materials, producing organic coatings to meet requirements for global war end uses. Because of their fundamental chemical and engineering training and their solid technical background experience, these technical men solved practically all the problems successfully.

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[1] Read on June 26, 1946, before the Forty-Ninth Annual Meeting of the American Society for Testing Materials, Buffalo, N. Y.

[2] Vice-President and Technical Director, Hilo Varnish Corp., Brooklyn, N. Y.

In the case of the shifting availability of raw materials, the most acute of the three problems, these men had to meet current changes by using whatever materials were available. Time and again they had worked out a substitute for a necessary ingredient, and then were forced by fresh shortages to devise a substitute for the substitute. Very often the substitute had become an alternative raw material for the same end uses.

TABLE I.—COMPARATIVE COMPOSITIONS OF TYPICAL FATS AND OILS (**29**).

Acids	Double Bonds	Linseed Oil		Tung Oil	Perilla Oil	Soybean Oil	Safflower Oil
Stearic	0	5.2	5.2	1.2	7.2	4.2	1.5
Oleic	1	14.7	9.9	13.6	3.7	32.0	24.6
Linoleic	2	39.4	40.7		41.9	49.3	63.0
Linolenic	3	35.7	39.2	....	41.7	2.2	0.14
Eleostearic	3	....	....	72.8	....		..
Palmitic	0	....	....	3.7		6.5	3.9
Myristic	0	....	....	....	....	....	0.04
Arachidic	0	....	....			0.7	0.4
Glyceryl radical		4.6	4.6	4.4	4.6	4.6	4.6
Iodine number		182.9	190.7	217 real 165 Wijs	204	124–143	149

Acids	Double Bonds	Walnut Oil	Fish Oil (Menhaden)	Castor Oil	Olive Oil	Coconut Oil	Oiticica Oil
Stearic	0	0.9	....	2.5	2.3	2.8	10.7
Oleic	1	16.9	24	7.0	84	5.7	5.9
Linoleic	2	69.7	19	3.5	4.6	0.9	..
Linolenic	3	3.1	....	....	....	....	
Licanic	3	....		....	....	....	78.2
Clupanodonic	5	....	17.2	....		....	
Ricinoleic	1	....	....	80.0			
Palmitic	0	4.4	10.5	....	6.9	7.2	
Myristic	0	0.01	7.7	....	trace	18.0	....
Lauric	0	....	....		....	50.0	....
Capric	0	....	....	....	....	4.2	....
Caprylic	0	....	....	....		9.1	..
Caproci	0	0.01	....		0.1	trace	..
Dihydroxystearic	0	....		1–2	....		..
Palmitoleic	1		16.3				
Glyceryl radical		4.6	4.6	4.6	4.8	5.4	5.2
Iodine number		132–159	130–160	80–90	77–95	8–10	150

Conservation of raw materials was the second acute problem. Certain materials were impossible to get, except for rigid military uses. Certain other materials could be had, but in greatly reduced quantities, and replacement was not certain. They had to be used to the best advantages or, in some cases, not used at all but reserved for the most acute needs.

Producing organic coatings to meet requirements for global war end uses was a very large task. Lieutenant-Colonel Totten (**50**)[3] has stated that this was due both to the extreme severity of the conditions to which most of the finished articles were subjected and to the limited supply or actual unavailability of a great many of the raw materials required to produce the most durable finishes. Part of the equipment manufactured to fight the global war was used under tropical conditions where a relative humidity of 95 per cent and a temperature of 120 F. was usual, not unusual. Some of it was subjected to very rough handling during loading, transportation, and unloading; and some of the equipment was immersed in sea water for several days during landing operations. These conditions were much more stringent than those to which normal peacetime finishes are subjected. The knowledge and experience acquired in development of protective coatings for these more stringent conditions will be applied towards the production of similar civilian coatings.

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[3] The boldface numbers in parentheses refer to the references appended to this paper, see p. 590.

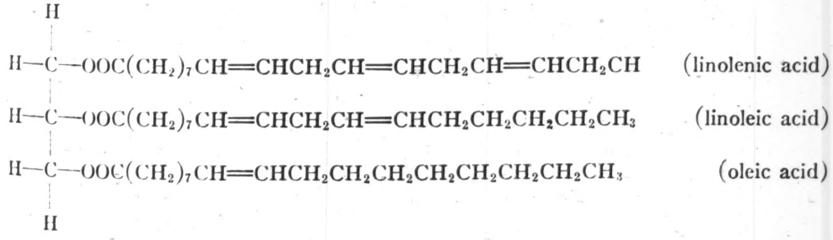
RAW MATERIALS

The initial physical, chemical, and subsequent processing and curing properties of the chemical raw materials are the first means of controlling the properties of the final dried coated films and the life or durability of coatings. The application of physical, chemical, and engineering principles to research and development of the raw material has been largely responsible for the chemical and engineering trend of the protective and decorative coating industry.

DRYING OILS

The drying oils (**29**) are mixtures of mixed triglycerides of unsaturated fatty acids. Most of the acids are 18-carbon acids and contain one, two, or three double bonds, although a small percentage of the acids in fish oil has four double bonds and some of them have a higher number of carbon atoms, up to 24.

On the same glycerol molecules there may be three different chains, or all three chains may be the same acids. In general, however, the three chains on the same glycerol molecules are different. The following formula may be considered as representative, indicating the general composition of the typical triglycerides found in linseed oil:



$$\begin{array}{ll} \mathrm{H} & \\ | & \\ \mathrm{H{-}C{-}OOC(CH_2)_7CH{=}CHCH_2CH{=}CHCH_2CH{=}CHCH_2CH} & \text{(linolenic acid)} \\ | & \\ \mathrm{H{-}C{-}OOC(CH_2)_7CH{=}CHCH_2CH{=}CHCH_2CH_2CH_2CH_2CH_3} & \text{(linoleic acid)} \\ | & \\ \mathrm{H{-}C{-}OOC(CH_2)_7CH{=}CHCH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_3} & \text{(oleic acid)} \\ | & \\ \mathrm{H} & \end{array}$$

The drying oils differ from one another in the proportions of the various 18-carbon acids and, in some few cases, of the higher carbon acids. By this difference the various drying oils are distinguished. Table I gives typical analyses of a number of the more common of them.

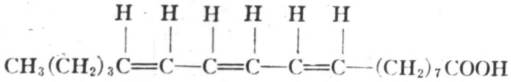
The composition varies, depending upon the climate, soil, particular crop year, date of harvesting of the seed, and other conditions. The figures given in the table, therefore, are not to be taken as strictly accurate ones to be remembered. It would probably be logical to round them off. For example, the composition of linseed oil could be remembered for discussion as:

5	per cent stearic acid
15	per cent oleic acid
40	per cent linoleic acid
35	per cent linolenic acid
5	per cent glyceryl radical

In contrast to this, soybean oil has much higher percentage of oleic acid, a little more linoleic, a very small percentage of linolenic acid, etc.

Two of the normally abundant naturally-occurring oils, namely, tung (China wood) and oiticica, differ from the others in that the double bonds in the 18-carbon chains are in what is called the conjugated position. This position is indicated by the following formula, which is the formula for eleostearic acid. Tung

(China wood) oil contains 70 per cent or more of this:



$$CH_3(CH_2)_3\overset{\overset{H}{|}}{C}=\overset{\overset{H}{|}}{C}-\overset{\overset{H}{|}}{C}=\overset{\overset{H}{|}}{C}-\overset{\overset{H}{|}}{C}=\overset{\overset{H}{|}}{C}-(CH_2)_7COOH$$

Following are the formulas written out for a number of the common acids encountered in the drying oils, showing the number of double bonds and their positions along the length of the chain:

FORMULAS OF ACIDS

Stearic: $CH_3(CH_2)_7CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2CH_2COOH$
Oleic: $CH_3(CH_2)_7CH=CH(CH_2)_7COOH$
Linoleic: $CH_3(CH_2)_4CH=CHCH_2CH=CH(CH_2)_7COOH$
Linolenic: $CH_3(CH_2)CH=CHCH_2CH=CHCH_2CH=CH(CH_2)_7COOH$
Eleostearic: $CH_3(CH_2)_3CH=CH-CH=CH-CH=CH(CH_2)_7COOH$
Licanic (oiticica oil): $CH_3(CH_2)_3CH=CH-CH=CH-CH=CH-(CH_2)_2CO-(CH_2)_4COOH$
Clupanodonic: $CH_3(CH_2)CH=CHCH_2CH=CHCH_2CH=CHCH_2CH=CHCH_2CH=CH(CH_2)_5COOH$
Ricinoleic: $CH_3(CH_2)_5CHOHCH_2CH=CH(CH_2)_7COOH$
Palmitic: $CH_3(CH_2)_{14}COOH$
Myristic: $CH_3(CH_2)_{12}COOH$
Lauric: $CH_3(CH_2)_{10}COOH$
Capric: $CH_3(CH_2)_8COOH$
Capryllic: $CH_3(CH_2)_6COOH$
Caproic: $CH_3(CH_2)_4COOH$
Arachidic: $CH_3(CH_2)_{18}COOH$
Dihydroxystearic: $CH_3(CH_2)_7CHOHCHOH-(CH_2)_7COOH$
Palmitoleic: $CH_3(CH_2)_7CH=CH(CH_2)_5COOH$
Glycerol: $CH_2OH-CHOH-CH_2OH$

Tung (China wood) oil, considered by many to be the king of the drying oils, at least for industrial uses, was available only in limited quantities, even for war requirements, during the war. The stoppage of shipment of tung oil from China gave great impetus to the development and production of chemically treated, isomerized, chemically reoriented, and synthetic drying oils. The underlying purpose was to produce quicker and harder drying oils with properties similar to tung oil. Considerable effort has also been expended in improving the natural oils in this direction.

Linseed oil was among the first drying oils employed in paints, and apparently was used alone as a protective coating. It still is the oil used in largest volume, but its future may be affected by the newer drying oils.

The first, though indirect, improvement of linseed oil has been through plant breeding. Flax strains are being developed richer in linolenic acid, which, being an unsaturated fatty acid with three double bonds, tends to give faster bodying drying oils.

The second improvement has been the use of additives which greatly influence the bodying speed of natural oils. The bodying speed of linseed oil, for instance, has been increased from 30 to 50 per cent, and bodied oils of lighter color and lower acid number have been produced by these catalysts.

The maleic—chemically treated oils have imparted to the nonconjugated oils the cooking speed of the conjugated type and give faster drying and greater hardness to varnishes in which they are used. This type of oil has replaced tung oil satisfactorily in many varnishes. The maleic oils are one example of increasing the functionability of the resulting oil molecule. Here the addition product of maleic anhydride and fatty acids, that is, linseed or soybean oils, can be esterified with glycerol, pentaerythritol, polypentaerythritols, or other polyhydric alcohols to yield a variety of drying oils of high molecular complexity.

Segregated oils are produced by a solvent-extraction process which can be carried out on either raw or bodied oils.

at present mainly linseed, fish, and soybean—either raw or bodied. These oils can be segregated into several fractions, one whose heat-bodying and drying tendencies are considerably faster and another whose bodying and drying tendencies are slower than the original oils. The former are especially adapted for use with dehydrated castor oil, since they tend towards brittleness and may need plasticizing. They are most valuable in the manufacture of hard, fast-drying varnishes.

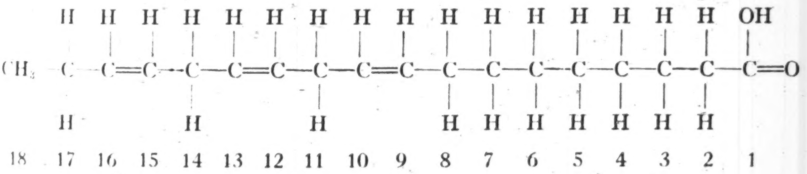
Split, distilled, and re-esterified linseed oil types are of current interest. The various esters of polyhydric alcohols and polyhydric alcohol polymers are very interesting for their fast polymerization and very good alkali resistance. Laboratory developments which show promise of improving linseed oil with respect to fast drying, alkali resistance, etc., involve the use of copolymers of linseed oil with such monomers as styrene, methyl methacrylate, butadiene, etc.

Oiticica oil (a nut oil, a native of Brazil) is the first natural oil to be used as a replacement for tung oil. It is of the mixed glyceride type. In contrast, tung oil is mainly a triglyceride of eleostearic acid. Both oils contain a triply conjugated, unsaturated fatty acid, but the acid of oiticica oil also contains a keto group. It has been used in some speciality finishes usually made with tung oil.

Dehydrated castor oil is the first chemically treated oil used extensively as a replacement for tung oil. When heat treated, it bodies several times faster than linseed oil and has found a permanent place in many hard-drying varnishes. It is fortunate that this oil was available to us during the war period. It is interesting to note briefly the chemical development of castor oil to the dehydrated oil.

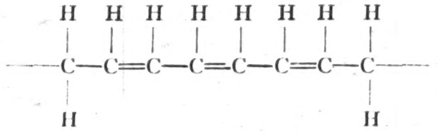
Castor oil is not a drying oil, but by dehydration process it is chemically converted into one. Through the work of Scheiber (46), it has been known for some years that the elimination of the elements of water from castor oil (dehydration) opens up a second double bond in each of the three ricinoleic acid radicals and thus produces an oil with good drying properties. In considering this improved drying property, first compare the two fatty acids linolenic and eleostearic:

Linolenic acid:



$$\begin{array}{cccccccccccccccccc}
 & H & H & H & H & H & H & H & H & H & H & H & H & H & H & H & H & OH \\
CH_3 & C- & C= & C- & C- & C= & C- & C- & C= & C- & C- & C- & C- & C- & C- & C- & C- & C=O \\
 & H & & & H & & & H & & & H & H & H & H & H & H & H & \\
18 & 17 & 16 & 15 & 14 & 13 & 12 & 11 & 10 & 9 & 8 & 7 & 6 & 5 & 4 & 3 & 2 & 1
\end{array}$$

Eleostearic acid:



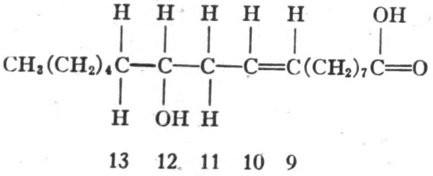
$$\begin{array}{cccccccc}
H & H & H & H & H & H & H & H \\
-C- & C= & C- & C= & C- & C= & C- & C- \\
H & & & & & & & H
\end{array}$$

In the linolenic acid the double bonds are at the 9-, 12-, 15-carbon atoms separated by a $-CH_2-$group; in the eleostearic acid they are at the 9-, 11-, 13-carbon atoms.

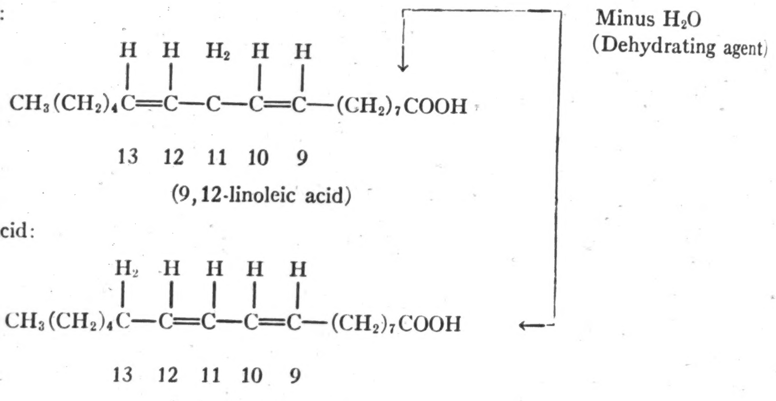
When ricinoleic acid is dehydrated,

both types of configuration are possible, as illustrated below:

Ricinoleic acid:


$$CH_3(CH_2)_4\underset{H}{\overset{H}{C}}-\underset{OH}{\overset{H}{C}}-\underset{H}{\overset{H}{C}}-\overset{H}{C}=\overset{H}{C}(CH_2)_7\overset{OH}{C}=O$$

Linoleic acid:


$$CH_3(CH_2)_4\overset{H}{C}=\overset{H}{C}-\overset{H_2}{C}-\overset{H}{C}=\overset{H}{C}-(CH_2)_7COOH$$
(9,12-linoleic acid)

Conjugated acid:

$$CH_3(CH_2)_4\overset{H_2}{C}-\overset{H}{C}=\overset{H}{C}-\overset{H}{C}=\overset{H}{C}-(CH_2)_7COOH$$
(9,11-linoleic acid)

On the practical basis of producing dehydrated castor oil, both types of these conjugated and nonconjugated acids are formed and about 15 per cent of other acids. On the basis of these properties and actual test, dehydrated castor oil lies between linseed oil and tung oil.

When heat treated, it bodies several times faster than linseed oil and has found a permanent place in many hard-drying varnishes.

Isomerized oils (25), drying oils known to contain conjugated unsaturated fatty acids, dry more rapidly and use less aerial oxygen during the drying process. In many natural oils, isolated double bonds predominate, and efforts have been directed toward transforming the isolated bonds to conjugated ones. Isomerized linseed and soybean oils are produced commercially. As would be expected, they have the characteristics of tung oil; for example, alone they dry to a matte or frosted finish. They have greater speed of drying and greater water resistance, properties which are also exhibited in the resulting varnishes. Isomerized oils also body more rapidly than the original oils. The development of these oils by re-esterifying the conjugated fatty acids with higher alcohols other than glycerol has proceeded with considerable success.

Petroleum Drying Oils:

Highly unsaturated hydrocarbon oils derived from petroleum are available from a number of sources. These have drying properties, but have been employed as extenders for linseed oil and have been used in various paint products, principally in architectural finishes where outdoor durability is not too important. Considerable research is being conducted in this field, and oils with improved drying properties will be produced.

Fish oils, particularly menhaden, sardine, and pilchard, are used in large quantities in the raw condition, in the bodied state, and chemically treated.

Garcia nutans oil (39), which has only recently come to the attention of the industry, is considered to be a supertung superhard quick-drying oil. It is a nut oil, a native of Venezuela also cultivated in Mexico. Laboratory tests of paints and varnishes made with this oil give great promise. It is not now available commercially.

Sorbitol or Mannitol Oils:

Synthetic oils obtained by esterifying sorbitol with drying oil acids, have shown good performance in varnishes.

Tall oil, a waste product from the kraft paper process, has found a place in the coating industry. In its crude state it is a dark, bad smelling, nondrying liquid. It contains some fatty acids of drying oils, as well as rosin acids. It can be refined and improved for use in place of some of the critical oils of a drying vehicle. Tall oil can also be esterified with polyhydric alcohols and is used in alkyd. During the war, it was utilized to prepare driers in place of the critically needed naphthenates.

General Remarks:

The idea of building oil molecules of greater complexity in order to get better speed of drying, increased bodying under heat, and better alkali and water resistance will continue to receive the attention of chemists. This general approach was especially influenced by the ideas of functionality expressed by Kienle (27). The future trend points definitely towards the synthesis of specially prescribed esters and compounds derived from various fatty acids, polyhydric alcohols, and additive compounds to produce quick-drying oils and other compounds with film-drying properties.

RESINS

Rosin:

The naval stores industry provided the protective coating industry with the first resin, rosin, and the first thinner, turpentine, for paints. The gum naval stores industry, so-called, is extremely interesting. Its products are mainly rosin and turpentine, obtained from three different species of pine trees native to most of our Southern states. On the other hand, wood naval stores, much more recent in origin, consist of rosin, turpentine, and pine oil obtained not from living trees but from the stumps, roots, and top wood of dead trees. This industry is enjoying industrial progress because of the greater application of chemical and engineering principles to naval stores products. The three main significant trends are: the generation of toughness in rosin derivatives as contrasted with the brittleness and relative friability of esters and salts of rosin that have been known in the past; the further purification of pine resin acids; the conversion of rosin acids into primary alcohol, for example, hydroabietyl alcohol from rosin.

Rosin gave us the first so-called synthetic resin, ester gum (glycerol triabietate), which was used with tung oil in spar varnishes before the advent of phenolic resins. The next most important rosin ester is the pentaerythritol rosin ester and a number of modified resins based on it. By esterifying rosin with pentaerythritol instead of glycerol, two outstanding differences are achieved: an increase of some 25 C. in melting point and greater stability to heat. Analogous varnish films made with this resin, in comparison with ester gum, dry more rapidly and show better resistance to water and alkali. A possible explanation of improved properties is the development of a more complex resin molecule with pentaerythritol, of greater functionality than with glycerol. The pentaerythritol tetraabietate resin and its modification were used in Federal Specification varnishes and enamels; in Maritime Commission oleoresinous finishes; in enamels for corn and bean packs; in specification finishes for gun mounts, tanks, and trucks; in varnish-base adhesives for sticking glass fiber insulation to ship hulls; in concrete airfield runway marking paints, in flameproof tent duck finishes; in corrosion-resistant enamels for maintenance of powder plant machinery; in locomotive finishes; and in many other essential coatings.

Other rosin ester gum derivatives used extensively in varnishes are maleic-modified ester gum and phenolic-modified ester gum. The pentaerythritol esters are similarly modified; they require smaller amounts of maleic or phenolic derivatives to achieve given melting points than the glycerol esters.

Phenolic Resins:

The several classes of resins falling under the general heading "phenolic" remain today the outstanding raw materials of the coatings industry when durable protection is required, especially in the presence of moisture or deleterious atmospheres and environments. Where rigid specifications need to be met, the formulator generally turns to these resins.

Classes.—The so-called phenolic resins may be divided into the following seven principal classes, based on composition and properties.

1. *100 per cent phenolic, oil-soluble, non heat-hardening.*—This type of resin, based on paraphenyl phenol, for use with drying oils, was introduced to the industry in 1928. Up to that time the unreduced phenolic resins were not compatible with drying oils and were not used by the varnish maker. These coatings dried largely by polymerization rather than oxidation, and thus gave quick-drying films of greater durability and resistance to moisture and chemicals. Later other substituted phenols were used, such as *p*-tertiary butyl and amyl phenols. The advent of these 100 per cent phenolics for air-drying coatings made a sweeping change to more rigid specifications for spar varnishes, anti-corrosive metal primers, and chemical-resistant paints.

2. *100 per cent phenolic, oil-soluble, heat-hardening.*—These resins, introduced in the early 1930's, retained their heat reactivity or ability to polymerize and harden with heat, even when combined with varnish oils or blown oils. Thus they are used especially in coatings and insulating varnishes of the baking type. It was early discovered also that they may be heated with rosin, ester gum, or ester resins, and thus produce much higher melting points and resistance properties.

3. *100 per cent phenolic, oil-insoluble, heat-hardening, alcohol-soluble.*—This is the type of phenol-formaldehyde resin known since 1908 and largely used in molding and laminating. Although incompatible with drying oils in the usual varnish formulations, it is used in alcohol solution as a baking lacquer. By thus polymerizing with heat, it produces an infusible and insoluble finish for metal. Recently through control of the reaction it has been found possible to polymerize such a resin at lower temperatures, thus making it applicable to baking coatings for wood, as in furniture finishes.

4. *100 per cent phenolic liquid resin concentrates ("phenolic resin syrups").*—Available also to the varnish manufacturer is a group of phenolic concentrates in the form of syrups, not generally soluble in oils but used to reinforce rosin, fatty acids, and other resins. Thus by incorporating 5 to 25 per cent of the phenolic syrup, rosin or fatty acids, etc., may be modified to

produce higher melting points, quicker drying, and increased resistance properties.

5. *Reduced phenolics, resin-modified and oil-modified.*—Resin - Modified.—Rosin or ester gum modified with 5 to 15 per cent phenol-formaldehyde made their advent as varnish resins about 1924. They produced quicker drying than the natural resin and were used in the first so-called 4-hr. coatings. The use of the large percentage of rosin acids made the phenol-formaldehyde oil-soluble.

Oil-Modified.—About 1928 it was found that phenol-formaldehyde resin could be dispersed, by special procedure, in certain drying oils, such as tung oil, to give more concentrated phenolic content in a tough, flexible resin. The varnish manufacturer further dispersed and polymerized this resin in drying oils to produce longer oil varnishes and vehicles, especially of the baking type. Or the oil-modified resin could be polymerized further by heat to produce baking coatings, or used to modify nitrocellulose lacquers.

6. *Oil-reacted, completely polymerized phenolics.*—When colloidally dispersed in thinners, these are "dispersion resins." Another advance in the resin art was the appearance of a completely polymerized oil-modified phenolic resin, dispersed in thinners. This type of vehicle dries quickly by solvent evaporation, and because it requires no oxidation in film formation yields extremely durable and solvent-resistant finishes. This reacted resin-oil polymer results in excellent adhesion to metal and alloys.

7. *Phenolic-base resins for nitrocellulose lacquers.*—A special group of modified phenolics is used with nitrocellulose to produce marproof lacquers. Such films, because of the highly polymeric nature of the resin, have hardness, "cold-check" resistance, and the ability to withstand alcohol and other solvents.

Alkyd Resins:

The classical definition for alkyd resins is that they are the reaction products of polybasic acids and polyhydric alcohols. Such products have been known academically since Berzelius in 1847 made the glyceryl ester of tartaric acid and noted that it was a viscous, gummy mass. In 1901 Watson Smith made glyceryl phthalate, and many attempts were made to use it in plastics. However, these resinous materials were of no interest to the surface coating industry until they were modified with fatty acids. About 1914 the nonoxidizing fatty acids were combined with glyceryl phthalate and were of interest as plasticizers for nitrocellulose lacquers. Kienle in 1927 disclosed another important development covering the use of oxidizing fatty acids as modifiers, resulting in air-drying alkyds.

Phthalic anhydride has been by far the most important polybasic acid and glycerol the most important polyhydric alcohol used in alkyd resins, but either or both may be replaced in part or entirely by such acids as maleic, succinic, adipic, and sebacic, or by such alcohols as the glycols, pentaerythritol, and sorbitol. The type and amount of fatty acid modifier may also be varied over a wide range, and rosin or other natural and synthetic resins may be added. Therefore, the ingenuity and skill of the chemist and engineer will be recognized as combining to produce a very wide variety of extremely useful alkyd resins for organic coatings. The fatty acid modifiers are, of course, esterified in the process of resin manufacture, and such resins are, therefore, referred to as oil-modified alkyds. Other resins that are added are referred to as resin- and oil-modified alkyds. With phthalic alkyds, the practical limits for phthalic anhydride content are 25 to 50 per cent. With 25 to 35 per cent content they are usually soluble in aliphatic hydrocarbons, but with 35 to 50 per cent they require stronger solvents, such as the aromatic hydrocarbons. The oxidizing or air-drying types may be used alone as the sole film-forming constituent of clear or pigmented coatings or in combination with other film formers such as oils, amino resins, and cellulose derivatives. The nonoxidizing alkyds are used as plasticizers for nitrocellulose lacquers, amino resin baking finishes, and leather and textile finishes.

Replacement of phthalic anhydride with maleic increases the hardness and reduces the air-drying and baking time. Replacement with the straight chain acids, adipic or sebacic, produces softer, more flexible resins. Replacement of the glycerol with alcohols having greater functionality, such as pentaerythritol, also reduces the air-drying and baking time whereas replacement with the glycols produce nondrying, very flexible resins. Addition of rosin or other resins usually reduces the drying time but detracts from durability. Consequently resin-modified alkyds are used chiefly for interior finishes.

The excellent durability of oil-modified alkyds was clearly demonstrated on all types of equipment used by the Armed Forces during the war. For air-drying colored finishes, the various drying oil acids, linseed, tung, dehydrated castor, etc., may be used, but for better color and color retention with age the soybean oil type acids are used. Finishes for automobiles and gasoline pumps, floor and deck enamels, and architectural white finishes may be made which air-dry in 4 to 8 hr. or bake in 1 to 2 hr. at 200 to 250 F. The high-speed production of modern industry requires very fast drying schedules so that baking finishes are used wherever possible. This emphasizes the added advantage of the use of nondrying alkyds which have extremely pale color and nonyellowing properties. In combination with the water-white amino resins, white baking refrigerator finishes are possible which will bake in 30 min. at 300 F., having color, hardness, toughness, and chemical resistance which would have been impossible a few years ago. Further details of these alkyd-amino resin finishes are given under the section on amino resins.

The many variations of the very flexible so-called glycol-sebacic type alkyds are widely used in combination with nitrocellulose to produce coatings on textiles for imitation leather, hospital sheeting, baby pants, shower curtains, or for the protection of insulation on electric cables. In combination with other cellulosic materials, synthetic rubbers, vinyl resins, and other polymeric film formers, the plasticizing alkyds will find wide range of industrial coatings.

Amino Resins:

The commercial development of amino resins provides another illustration of the enlarged possibilities for protective coatings from the use of synthetic materials. Made from strictly chemical compounds, the amino resins have characteristics which cannot be approached by any of the natural resins. Urea and melamine, the principal amino compounds used, are condensed with formaldehyde to form the primary reaction product. This product may be alkylated with a variety of alcohols to form ether-type compounds having a range of solubilities in such solvents as water, alcohols, or hydrocarbons. The various amino resins are used in many industries besides the surface coating industry. They are important in the plastics industry and have found unique uses in textile treatment, the manufacture of wet strength paper, special tannage for leather, and in the demineralization of water.

Urea was one of the first so-called organic compounds to be synthesized from inorganic materials. Wöhler's historic words, written in 1828, "I can now make urea without the need of kidneys," is generally considered to be the birth announcement of synthetic organic chemistry. His friend, von Liebig, in 1834 made another amino compound which he called melamine. It was, however, more than a century later before these materials were used in surface coatings. The commercial production of these chemicals had to await modern engineering materials and methods to be economical. The difference in structural formulas and physical characteristics of urea and melamine is illustrated in Fig. 1.

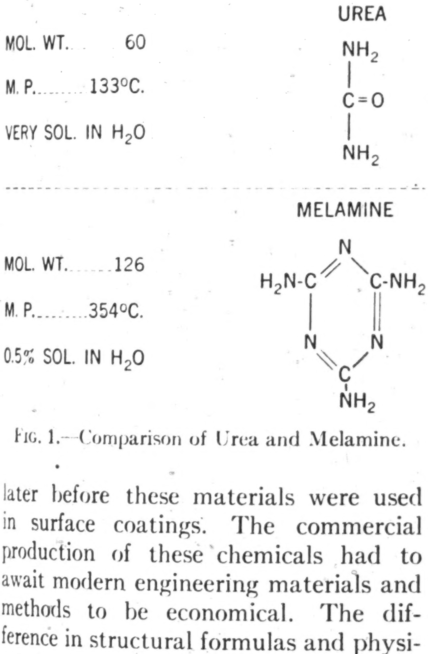


FIG. 1.—Comparison of Urea and Melamine.

Urea and melamine are believed to exist in tautomeric forms, and, when reacted with formaldehyde and alcohols, a number of reactions occur simultaneously so that the final product is a very complex compound.

Amino resins are thermosetting and are, therefore, used in baking finishes. They form clear, water-white, extremely hard films with good chemical resistance. In fact, they are so hard that they are commonly used in combination with softer resins such as the phthalic alkyd resins to obtain the required flexibility. They make an ideal combination because the amino resins impart a scratch-resistant surface and improved alkali resistance to the good qualities of toughness, adhesion, and durability of the alkyd resins. Amino resins also reduce the time necessary for baking the enamels to at most 50 per cent of that used previously.

Melamine resins are a more recent development than urea resins and are slightly higher in cost, but their better performance more than offsets the cost factor. In general they are faster curing, more heat stable, and are, therefore, used over a wider temperature range; they also have better chemical resistance than urea resins. In the baking range of 250 to 300 F., both urea and melamine resins may be used in amounts of 5 to 50 per cent, the remainder being alkyd resin. The melamine resins, however, may be used in a baking range of 200 to 350 F., and for short-time periods as high as 400 F. Typical samples of industrial baking finishes which give outstanding performance are:

(*a*) Automobile finish, using about 15 per cent melamine resin with 85 per cent oxidizing alkyd resin, in a wide variety of colors, and baking at 275 F. for 30 min.

(*b*) Washing machine finish, requiring high resistance to hot soap solutions, using 30 to 40 per cent melamine resin with 60 to 70 per cent oxidizing alkyd, and baking at 300 F. for 45 min.

(*c*) Refrigerator finish, using about 30 per cent of a combination urea-melamine resin and 70 per cent of non-oxidizing alkyd for maximum whiteness, and baking at 275 to 300 F. for 30 min.

METALLIC SOAPS AND DRIERS

Metallic soaps serve very useful purposes as suspension, flatting, grinding aids, driers, sealers, anti-fouling, waterproofing, and for developing low-luster clear coatings, etc. The driers, particularly the iron, lead, cobalt, manganese, and zinc, have been used as resinates, linoleates, and naphthenates for many years.

THINNER

Petroleum thinners of both the normal and high-solvency types and the coal-tar solvents played a significant part in war work. In the formulation of industrial finishes for specific war uses, the thinners were very carefully selected for solvency and, particularly, for speed of drying of the final coatings. The coal-tar solvents used previously in the coating industry had gone to war for other industries. Under the pressure of war demands, Government agencies had issued new specifications which permitted the use of high-solvency aromatic petroleum naphthas in place of coal - tar solvents. Producers of petroleum thinner say that they will produce tailor-made thinners at a later date.

With regard to lacquer solvents, no significant changes have occurred within the last several years.

PIGMENTS

Under the impetus of the war, pigment manufacturers continually improved their pigments. One improvement was the development of a zinc chromate whose water-soluble salts were greatly reduced. This improvement appears to increase corrosion inhibition and resistance to blistering.

Red lead is still widely used as a corrosion-preventive pigment. Metal protective paints, made with faster-drying vehicles in which the pigment is predominantly red lead, are providing outstanding long-time protection.

Luminescent Pigments:

Improvements have been made both in the brightness of the fluorescence and in the length of the phosphorescence.

Aluminum Pigments:

The trend in aluminum paint appears to be towards a ready-mixed product which is ready for use by the consumer. The leafing stability of aluminum pigment in ready-mixed aluminum paint has been definitely improved as a result of development work carried out by the pigment manufacturers in the past year or so. During the war, aluminum paint was used for essential applications where such properties as resistance to heat and moisture penetration were necessary.

PAINTS AND ENAMELS

Interior and exterior finishes which were modified or changed by war emergencies may find a permanent place in the coating industry.

Primerless Flats:

A considerable number of so-called primerless flats have appeared within recent years. They are a logical outgrowth of the calci-coaters and combine the easy application properties of emulsion paints with the washability of oil paints. They represent an improvement over calci-coaters in that they produce films which are more readily recoated when redecoration is in order, this property being achieved by a judicious selection of pigments and extenders combined with heavy, treated oils blended with suitable resins. These primerless flats are designed to produce a uniform finish over surfaces of widely varying porosity, and many of them do not fall far short of this goal.

Emulsion Paints:

The popularity of emulsion paints

has been well maintained during the past years. It is attributed mainly to the scarcity of oil, but undoubtedly the inherent application properties and relative freedom from objectionable odor have been important factors to the ultimate consumer. Improvements in washability have been effected to an extent sufficient to lead to a proposal for a more rigid performance requirement in Federal Specifications. These coatings have been produced to date of flat luster, and present indication is that gloss emulsion enamel will be successfully produced.

Outside House Paints:

House Paint Pigmentation.—The pigmentation trend in house paints seems well established toward lower lead and increased titanium dioxide. This trend can be observed by a careful analysis of the paints listed in the National Paint, Varnish and Lacquer Association *Circulars 648 and 669.* Such an analysis shows trends toward: (*a*) titanium dioxide-leaded zinc-extender and/or lead; (*b*) titanium-magnesium pigment and titanium - calcium pigment away from titanium-barium pigment; (*c*) lower lead in titanium-type pigmentations.

Bodied Oil.—House paints made with limited amounts of bodied linseed oil with low acid number seem well established as a result of the war. The performance of such conservation paints to date leaves little doubt that paints formulated with reasonable amounts of bodied linseed oil, together with somewhat higher than normal pigment volume and thinner content, will continue to command interest. In this connection means of obtaining good application properties are important and become more so as the viscosity of the bodied linseed oil used is increased. It is quite probable that application properties together with ultimate durability characteristics which generally are not yet known will determine to a very great extent the bodied oil content of the future house paints.

PLASTICS

The application of solid or fluid plastics in film form opens a whole new field of materials that can be used as protective coatings. These plastics are composed entirely of film-forming materials (100 per cent nonvolatile).

Polyethylene resins have favorable electrical characteristics for use in electronic application. Polyethylene plastics are tough, have good impact resistance and extremely low moisture vapor transmission, and unusually low water absorption. Their chemical resistance is outstanding. Polyethylene is one of the lightest plastics, so light that it will float in water. It remains usable at temperatures lower than 90 F. and is sufficiently rigid for use in temperatures up to 230 F. Polyethylene is colorless and translucent but can be prepared in color. The important point to emphasize is that polyethylene plastics can be flame-sprayed as protective coatings on metal surfaces, with resulting films of good adhesion, and, in addition, they exhibit the good chemical and physical properties already given for these resins. Other suggested uses for these resins are for washers, gaskets, and other molded articles, films for coated fabrics and papers, collapsible tubes, tubings, filaments, etc.

The general question of plastics *versus* organic coatings will be discussed later.

COATINGS

All organic coatings may be classified into two main groups on the basis of physical appearance: clear transparent films and opaque and colored films.

They may be designed as varnishes, clear lacquers, or clear coatings when used to produce a clear transparent film; and vehicles or binders when used to produce opaque and colored films.

VARNISHES

Definition:

The definition of a varnish, as given by A.S.T.M. Tentative Definition of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 - 44 T)[4] is: "A liquid composition which is converted to a transparent or translucent solid film after application as a thin layer."

There can be no doubt that varnishes are a heterogeneous system, consisting of resin and thinners, or resins, oils, thinners, and driers. Some consider varnish a solution; others classify it as a colloid. They are partly a solution and partly a colloid.

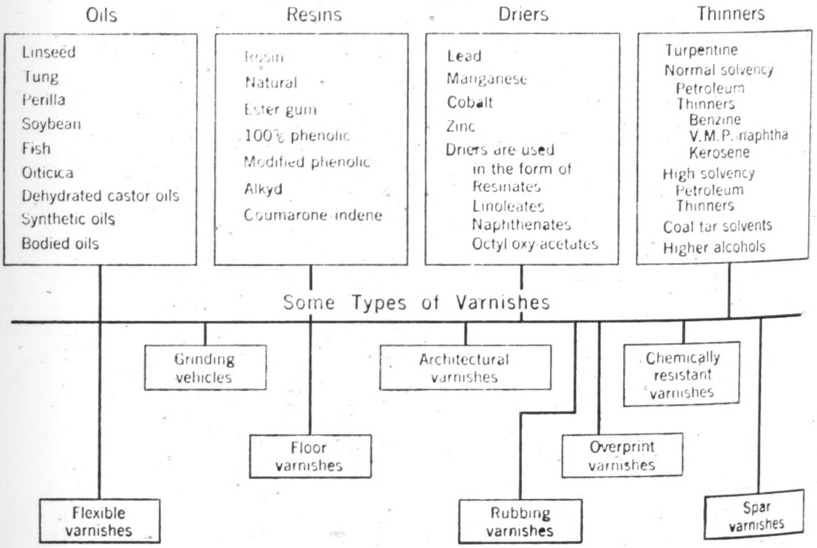
In a physical sense, varnishes are fluids that can be spread readily to thin smooth films which dry to hard, continuous, permanent, solid coatings when exposed to air or heat. The first step in the drying of varnish films is the evaporation of the volatile portion, which leaves the nonvolatile material to dry by oxidation and/or a combination of polymerization and condensation. Some varnishes dry only by evaporation—for example, cold-cut resin varnishes and shellac.

Classification:

According to their constituents, varnishes may be divided into two main types:

1. Oleoresinous varnishes
    - (*a*) Oil plus
        - (1) Natural resins
        - (2) Synthetic resins
    - (*b*) Oil
    - (*c*) Oil-modified
        - (1) Glycerol phthalate
        - (2) Pentaerythritol phthalate or other esters
        - (3) Chlorinated rubber

Table II.—
Composition of Oleoresinous Varnishes


Oils
Linseed
Tung
Perilla
Soybean
Fish
Oiticica
Dehydrated castor oils
Synthetic oils
Bodied oils
Resins
Rosin
Natural
Ester gum
100% phenolic
Modified phenolic
Alkyd
Coumarone indene
Driers
Lead
Manganese
Cobalt
Zinc
Driers are used in the form of
Resinates
Linoleates
Naphthenates
Octyl oxy-acetates
Thinners
Turpentine
Normal solvency
Petroleum
Thinners
Benzine
V.M.P. naphtha
Kerosene
High solvency
Petroleum
Thinners
Coal tar solvents
Higher alcohols
Some Types of Varnishes
Grinding vehicles
Architectural varnishes
Chemically resistant varnishes
Floor varnishes
Overprint varnishes
Flexible varnishes
Rubbing varnishes
Spar varnishes

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[4] 1944 Book of A.S.T.M. Standards, Part II, p. 1551.

2. Spirit varnishes
    (*a*) Solvent plus
        (1) Natural resins
        (2) Synthetic resins

Type 1 dries by oxidation, or oxidation and condensation, or by oxidation, condensation, and polymerization. Type 2 dries either solely by evaporation or by evaporation and some oxidation and/or polymerization.

Oleoresinous Varnishes:

General Composition.—The four main (Table II) constituents (raw materials) of the oleoresinous varnishes are:

1. (*a*) Drying and semi-drying vegetable oils.
    (*b*) Drying and semi-drying fish oils.
    (*c*) Oils chemically converted.
    (*d*) Synthetic drying oils.
2. Resins.
3. Driers.
4. Thinners (or solvents).

They are illustrated diagrammatically in Table II.

Function of Each Constituent.—The resins impart hardness, toughness, ductility, adhesion, waterproofness, and gloss. Hard resins accelerate drying.

The oils impart flexibility, toughness, adhesion, durability, and relative waterproofness.

The driers have, as their main function, the acceleration of the drying time.

The primary function of thinners is to reduce viscosity or increase fluidity so as to permit easy application of the varnish coating. Slow - evaporating solvents also have the effect of increasing the hardness of a film. When resins and oils are processed together (cooked), a very viscous substance is obtained which is difficult to spread into a thin film. In reducing the viscosity, the thinners or mixture of thinners used must satisfactorily dissolve or disperse all the solid ingredients of the varnish. This is true in general for all vehicles.

Characteristics of Oleoresinous Varnishes.—In designing a varnish, it is absolutely necessary to know the performance expected. For example:

1. *Floor varnish* must dry overnight to a tough, hard film of good gloss; must have good water and alkali resistance and good resistance to scuffing.
2. *Spar varnish* should dry to a hard, glossy film which must stand the atmospheric conditions prevailing: salt air, actinic rays of the sun, moderate chemical resistance, etc.
3. *Chemically resistant varnishes* must be designed to withstand acids, alkali, and other chemicals with which they may come in contact.
4. Many other examples, each to meet some special requirements.

Varnish "Oil Length."—A varnish may be composed of one of each of the components enumerated; however, most varnishes are composed of several resins, oils, driers, and thinners.

Varnishes are known as of short, medium, or long "oil length." The oil length is defined as the number of gallons of oil used per 100 lb of resin. Varnishes of short oil length will dry faster and will usually be more brittle than varnishes of long oil lengths.

TABLE III.

	45-gal. Varnish (Spar Varnish type)	
Basis:	100 lb. resin 45 gal. oil	Varies from 45 to 60 per cent nonvolatile
	x gal. or lb. drier y gal. thinner	Varies from 55 to 40 per cent volatile
	18-gal. Varnish (Floor Varnish Type)	
Basis:	100 lb. resin 18 gal. oil	Varies from 45 to 50 per cent nonvolatile
	x' gal. or lb. drier y' gal. thinner	Varies from 55 to 50 per cent volatile

Table III illustrates a 45-gal. varnish, spar varnish type, and an 18-gal. varnish, floor varnish type.

Testing.—Varnishes are usually examined by the manufacturer for appearance (clarity), color, viscosity, specific gravity, drying time, etc. With some varnishes, it is necessary to check their water, alkali, and acid resistance. Rubbing

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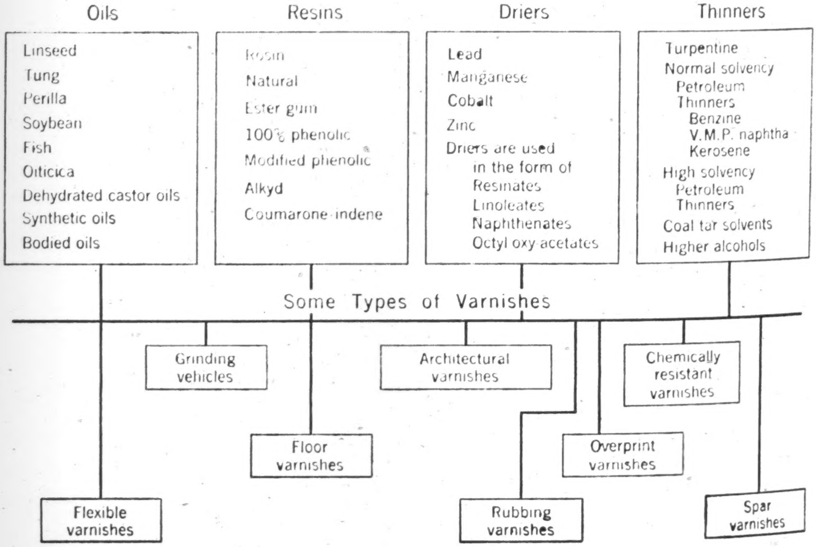
Oils
Linseed
Tung
Perilla
Soybean
Fish
Oiticica
Dehydrated castor oils
Synthetic oils
Bodied oils
Resins
Rosin
Natural
Ester gum
100% phenolic
Modified phenolic
Alkyd
Coumarone-indene
Driers
Lead
Manganese
Cobalt
Zinc
Driers are used
in the form of
Resinates
Linoleates
Naphthenates
Octyl oxy-acetates
Thinners
Turpentine
Normal solvency
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Basis:	100 lb. resin 18 gal. oil	Varies from 45 to 50 per cent nonvolatile
	x' gal. or lb. drier y' gal. thinner	Varies from 55 to 50 per cent volatile

Table III illustrates a 45-gal. varnish, spar varnish type, and an 18-gal. varnish, floor varnish type.

Testing.—Varnishes are usually examined by the manufacturer for appearance (clarity), color, viscosity, specific gravity, drying time, etc. With some varnishes, it is necessary to check their water, alkali, and acid resistance. Rubbing varnishes should be checked for rubbing and polishing; and insulating varnishes, for electrical insulating properties. Some varnishes may have to be resistant to specific reagents.

Application.—Varnishes are applied either by brushing (mainly for painting homes and buildings) or by spraying or dipping (mainly for commercial uses).

Uses.—Varnishes are used on surfaces either for interior or exterior exposure and on many hundreds of industrial articles.

LACQUER (NITROCELLULOSE)

Definition:

The word "Lacquer" is generally considered to indicate a liquid coating, made up of volatile and nonvolatile portions, which dries rapidly and leaves a hard tough film when applied in thin coats. Webster's Dictionary mentions four types of lacquer:

1. A spirit (alcohol used as solvent) varnish, as shellac, often colored, used especially for coating brass and other metals to heighten their luster or prevent tarnishing.

2. Japanese or Chinese lacquer, the chief constituent of which is urushiol, obtained by tapping the Japanese varnish tree, straining, and drying the sap by heat. To the resulting dark brown syrupy liquid are usually added diluents, pigments, etc. Burmese lacquer and Formosan lacquer contain thitsol or caccol in place of urushiol.

3. Any of the various artificial varnishes or varnish paints, usually colored and often opaque, in which pyroxylin, resins (natural and synthetic), ester gum, asphalt, cellulose acetate, etc., serve as bases.

4. An oleoresinous coating for food containers, usually hardened by baking.

Although the products first named were formerly known as lacquers, the present day lacquer refers primarily to nitrocellulose (pyroxylin) coatings, which may be either clear or pigmented.

Nitrocellulose lacquers dry primarily by evaporation, a fast process as compared with the relatively slower process of oxidation which causes the air drying of oleoresinous finishes.

Lacquers are also composed of four general components: resins, plasticizers, nitrocellulose (cellulose nitrate) or other cellulose esters, and thinners.

The thinners consist of a mixture of active solvents, latent solvents, and diluents.

Resins impart the following properties to nitrocellulose lacquers:

1. Increased adhesion, as nitrocellulose alone is notably lacking in this desirable characteristic.

2. Increased solid content, body, and depth, and with some resins without changing the viscosity appreciably or unduly raising the cost.

3. Increased gloss, luster and brilliancy. Improved outdoor durability by the use of certain alkyd resins, particularly in clear finishing lacquers. Improved water resistance and chemical resistance as may be necessary under special conditions.

Plasticizers (Softeners):

The plasticizers impart the elements of elasticity, durability, and adhesion. They are either solid or very slow-evaporating liquids. They improve the flowing properties of lacquers, inhibit blushing and enhance the luster of the dry film.

Nitrocellulose provides the element of toughness and assists in making a continuous film. The nitrocellulose solutions offered in the market vary in viscosity from $\frac{1}{2}$ sec. to 150 sec. These nitrocellulose solutions of different viscosities may be used to adjust the viscosity of the final lacquer. Viscosity, the most important physical property, is of great value in controlling the finished product.

Solvents, Latent Solvents, and Diluents:

The important function of these is solvency and the reduction of the viscosity of the viscous mass, which is composed of nitrocellulose, resin, and plasticizer, to that fluidity which will permit easy spreading of the lacquer to a thin film.

Use Requirements:

The performance of the finished lacquer governs the selection of resins and plasticizers, as well as the solvents and other ingredients. Adhesion of film is increased by the proper choice of resins or resins and plasticizers combined. Those resins which impart good adhesion to lacquers are shellac, elemi, alkyds, kauri, pontianac, congo, and zanzibar. In the formulation of lacquer, it is very often necessary to use several resins to get the desired properties such as body, gloss, adhesion, fullness of film, or other performance characteristics.

FUNDAMENTAL INVESTIGATIONS

Fundamental investigations have played a significant rôle in the protective coating field, as in other chemical and engineering industries, to establish a more scientific understanding of the chemical and engineering aspect of the industry. Examples of such investigations are those conducted by J. S. Long (**30**), T. F. Bradley (**5**), B. P. Caldwell (**11**), and J. J. Mattiello and L. T. Work (**34**) on the bodying of linseed oil.

Perhaps one of the most time-consuming operations in the manufacturing of paints and varnishes has been the preparation of bodied oils. Compared with other operations, it is costly.

During the process of bodying drying oils from the raw state to that of heavy viscous liquids, many chemical and physical changes occur concurrently. Although the nature of these changes and all the factors that affect their speed and sequence are not known, it is agreed that reactions of polymerization, condensation, cracking, oxidation, hydrolysis, rearrangement, and gelation are involved. The sequence of some of these reactions and other chemical changes that occur during bodying have been discussed by Long (**30**), Bradley (**5**), and others.

The art and skill of the old-time varnish maker have yielded only slowly to the more scientific control and to the developments of the chemists and engineers who have entered the coating materials industries. The complexity of the organic molecules which they have had to deal with, however, has necessitated the continued use of methods which, although more exact and more scientifically grounded than those of their predecessors, nevertheless are still quite empirical.

With the growth of the oil-processing and oil-consuming industries it became increasingly apparent that progress was largely circumscribed by the lack of adequate fundamental knowledge. On this account, during recent years, considerable support has been accorded the more fundamental investigations.

From a practical aspect the wetting and livering properties of the bodied oils for the various pigments used in the production of pigmented coatings are of vital importance. A comprehensive survey of the chemical and physical properties, such as viscosity, specific gravity, color, apparent mean molecular weight, index of refraction, acid number, or iodine number, determined at various stages of bodying, does not help in evaluating the wetting and livering properties of the bodied oils. Practical tests, the actual making of pigment oil pastes, are of considerable value for the purpose of standardizing and controlling reactions both for the bodying of the oil and evaluating pigment oil systems used in pigmented coatings.

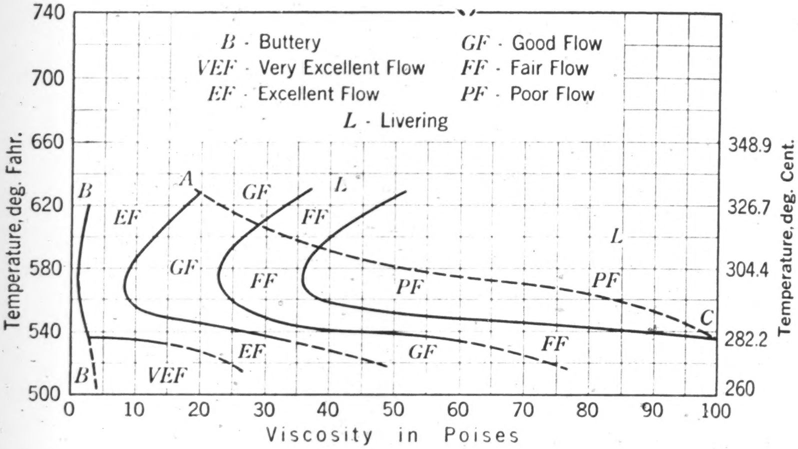

FIG. 2.—Flow and Livering Characteristics of Peacock Blue Pastes Made with Bodied Linseed Oils Processed in Air (**34**).

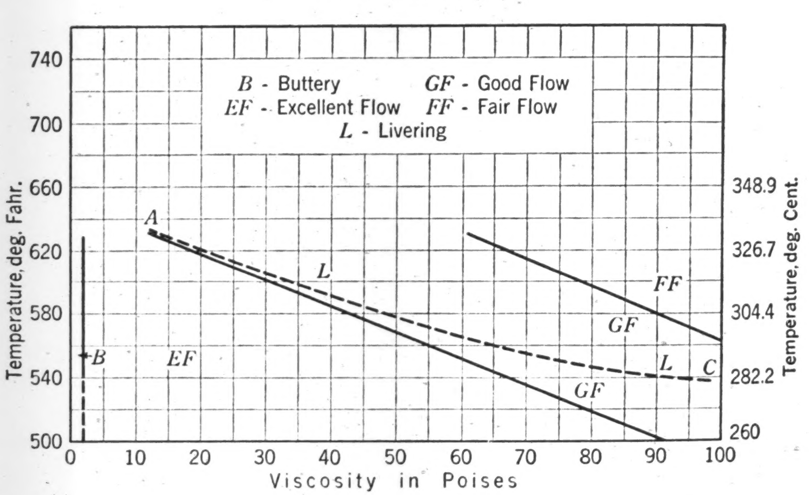

FIG. 3.—Flow and Livering Characteristics of Zinc Oxide Pastes Made with Bodied Linseed Oils Processed in Air (**34**).

The work of Mattiello and Work (**34**) is of interest in this connection. They investigated the wetting and livering properties of four pigments—peacock blue, zinc oxide, titanium oxide, and carbon blacks—with oils bodied at temperatures varying from 535 F. to 600 F. and exposed to air and under vacuum. Figures 2, 3, 4, 5, and 6 illustrate graphically the results of pigment oil pastes made with the bodied oils, ranging in viscosity from raw oil of 0.5 poise to very heavy bodied oils of 150 poises. The flow characteristics of the pastes were arbitrarily chosen and quantitatively defined by means of a Binney & Smith flowmeter (**34**) as having buttery consistency or as "very excellent, excellent, very good, good, fair, and poor flow."

These properties of the bodied oils for the pigments are a function of the bodying temperatures and viscosity of the bodied oil, shown graphically as flow areas in the figures just mentioned.

It is particularly interesting to note that for the first three pigments the wetting characteristics of the bodied oils improve considerably as the oil increases in viscosity from raw state to about 5 poises. Thereafter the improvement in wetting properties is much less as the viscosity of the oil increases.

The development of the livering area which is above the line *A-C*, Figs. 2, and 3, is of considerable practical importance.

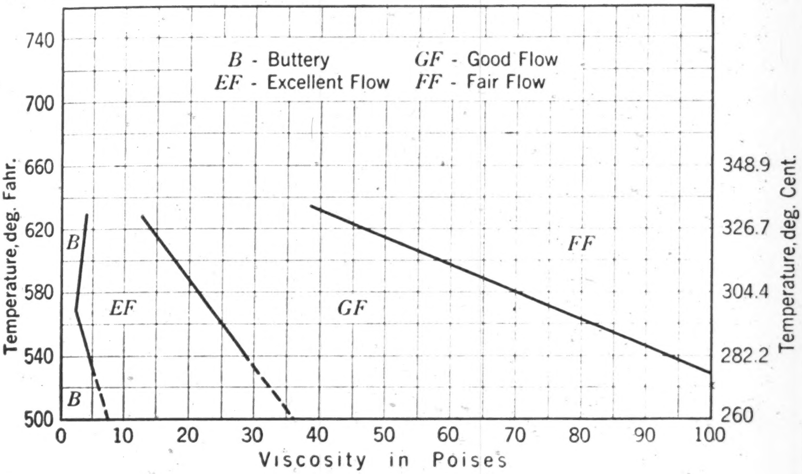

FIG. 4.—Flow Characteristics of Titanium Oxide Pastes Made with Bodied Linseed Oils Processed in Air (34).

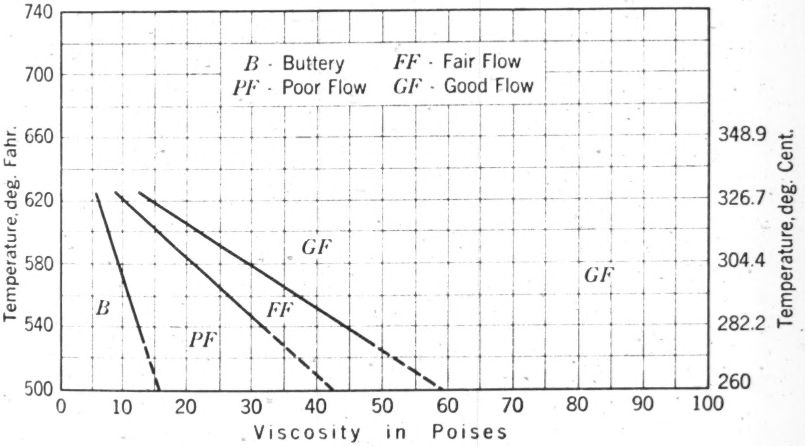

FIG. 5.—Flow Characteristics of Carbon Long-Type Black Pastes Made with Bodied Linseed Oils Processed in Air (**34**).

Livering is the term applied to a state of fixed rigidity of a pigment-oil paste which results from a drastic change in consistency upon aging. This change may be of such magnitude that pastes of excellent, good, or poor flow may alter to a rigid state. The phenomenon of livering is the result of an irreversible reaction. The illustration presented clearly indicates how to process bodied oils with nonlivering tendency.

It is particularly interesting to note the viscosity of the bodied oils which just changes the consistency of the pastes from buttery consistency to either very excellent or excellent flow. These values place the pigments discussed in the following order of diminishing ability of the bodied oils to wet the pigments.

1. Zinc oxide.
2. Peacock blue.
3. Titanium oxide.
4. Carbon black—long type.
5. Carbon black—short type.

A bodied oil in comparison with raw oil is also improved considerably in its ability to dry more quickly, in its resistance to water and to alkali, increased impermeability to moisture, and in ability to wet pigments easily. A bodied oil has less tendency to penetrate and, therefore, produces a dried film of higher luster. A bodied oil has better binding properties for pigments; on the other hand, when used in pigmented products (paints and enamels), a bodied oil induces hard brushing because of its increased tack.

This kind of investigation extended to house paints proved of extreme value during the war period because of scarcity of linseed oil.

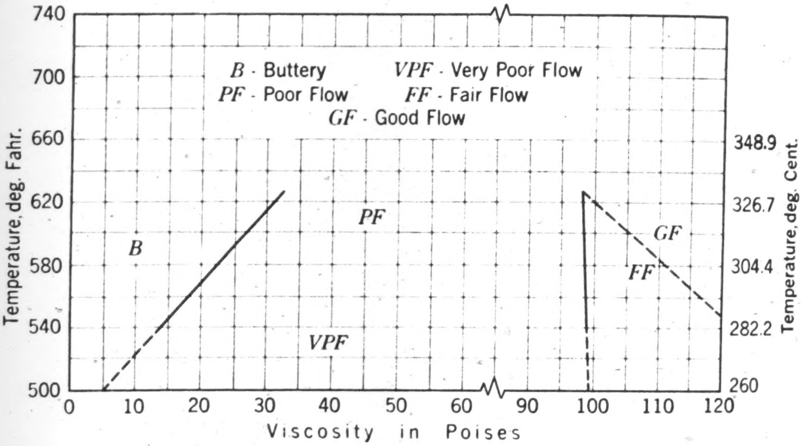

FIG. 6.—Flow Characteristics of Carbon, Short-Type Black Pastes Made with Bodied Linseed Oils Processed in Air (**34**).

BODIED LINSEED OILS IN OUTSIDE HOUSE PAINTS

The formulation of outside house paints over the past twenty years has seen many changes in the composition of the pigment. The vehicle (called standard), however, remained practically unchanged as: 90 per cent binder and 10 per cent thinners and driers; the binder consisting of about 92 per cent raw or refined linseed oil and 8 per cent air-bodied linseed oil of about Q (4.35 poises) viscosity.

About eight years ago an exposure program was started with the specific plan of developing improved house paint finishes through the use of bodied or processed oils. These first studies included a number of drying oils other than linseed oil and several series of bodied oils.

Starting with an alkali-refined linseed oil, the oil was bodied at 600 F. and at 535 F. in both air and vacuum. The 600 F. air-bodied oils were bodied to viscosities ranging from 0.2 to 160 poises and the acid numbers of these oils tested were 0.28 to 15.9. The air-bodied oils bodied at 535 F. also ranged from 0.2 to 160 poises with acid numbers of 0.28 to 7.5. The oils bodied in vacuum at both 600 F. and at 535 F. ranged in viscosity from 0.2 to 250 poises and their acid numbers ranged from 0.2 to 2.5.

The standard vehicle already mentioned was used as the control. A pigmentation widely made commercially was used in all the paints. The bodied oils were used as 100 per cent of the binder; as 25 per cent of the binder, the remaining 75 per cent being alkali-refined linseed oil; and as 10 per cent of the binder, the remaining 90 per cent being alkali-refined linseed oil. The paints were all formulated at 28.5 per cent pigment volume, adjusting consistency with mineral spirits to standard Kreb's units as measured on the Stormer Viscometer. Kreb's units are given by Gardner (**22**, p. 306).

Four years exposure of these paints developed definite information which permitted the drawing of several interesting conclusions regarding the durability characteristics of the bodied oils—their resistance to loss of film integrity as compared to the standard vehicle which had been used for many years. The bodied oils were more subject to checking cracking failure. In the air-bodied oils, no noticeable drop in durability was noted with increasing viscosity up to about X (12.9 poises) viscosity. At about X viscosity there was a definite break in durability, the failures increasing in severity with increasing viscosity. This was true at about the same point—X viscosity—for both the series bodied at 600 F. and the series bodied at 535 F., though acid number increased more rapidly for the oils bodied at the higher temperature. For the vacuum-bodied oils again there was a break in durability at the higher temperature and at the higher viscosities. However, with the vacuum-bodied oils the break occurred at a viscosity somewhat higher than X; also the breaks were not as definite or as severe for the vacuum-bodied oils as for the air-bodied oils.

Apparently durability was impaired by both high viscosity and increasing acid number. From this work it was felt that air-bodied oils up to X viscosity could be used safely provided the acid number was not too high (in the range of 7.0 acid number as a maximum) and that with vacuum-bodied oils somewhat higher viscosities could be tolerated.

It was interesting to note that these results held fairly constant whether the bodied oil constituted 100, 25, or 10 per cent of the total binder. This indicated that even a comparatively small amount of improperly selected bodied oil was injurious to film durability.

About this time the demands of the war brought on a severe shortage of drying oils and plans were made to conserve linseed oil by reducing the amount permitted per gallon in various types of paints. House paints, as produced for many years, carried about 5 lb. of linseed oil per gallon. Conservation formulations were issued reducing the linseed oil content to 3.75 lb. per gallon, keeping the pigmentation unchanged and making up the lost volume with mineral spirits. This automatically raised pigment volume from 28.5 per cent to 35 per cent. To maintain paint consistency this could be accomplished by the use of bodied oils as all or part of the binder.

The original conservation formulations were written with 50 per cent of the binder as Z4 (63.4 poises) viscosity linseed oil—no limitations as to methods of bodying, acid number, etc. These paints were too heavy in consistency, requiring considerable reduction with mineral spirits before using. Even when further reduced, the paints had poor application properties.

Information from these bodied-oil studies made it apparent that use of Z4 viscosity linseed oil would be expected to lead to poor durability and to final films with poor repaint characteristics. These conservation formulations were soon revised without limitations as to the amount of bodied oil to be used or the viscosity of the bodied oil used. This revision permitted the paint technician to take advantage of the information available from these bodied-oil studies and to formulate their conservation paints around bodied oils of lower viscosities with reasonable assurance that good results should be obtained.

In these conservation paints, the chief unknown factor was the effect of increasing pigment volume from 28.5 per cent to 35 per cent. This change would be expected to lead to thinner films, though the drop in film thickness should be partly offset by the better nonpenetrating properties of the bodied oils. In unbodied-oil house paints, increasing pigment volume leads to more rapid chalking rates and resultant shorter film life. This cannot be corrected by the use of slower chalking pigments because of the increased dirt and mildew collection and poor appearance. It was expected that the bodied oils would show somewhat slower chalking and at least partly offset the increased chalking rates resulting from the increased pigment volumes.

To determine the effect of these various formulation changes, extensive exposure studies were undertaken. The results, now four years old, have given us these important conclusions: with proper control of the bodied oil and with pigmentations having good check-crack resistance properties, house paints of the general composition of the conservation formulations can be made to yield appearance, protection, and repaint surfaces comparable with the pre-war type house paints. They afford a substantial saving in linseed oil consumption.

With the pigment combination used in these studies we have drawn the following conclusions, based on four years south vertical exposures.

1. With 92 per cent alkali-refined linseed oil plus 8 per cent Q (4.35 poises) viscosity linseed oil:

Durability perfect at 28.5 per cent PV (pigment-volume percentage of total paint). Failures apparent at 32 per cent PV and increasing in severity at 35 per cent PV and at 40 per cent PV owing to increased chalking rate to thin films with resultant loss of resistance of the film to cracking, etc. Durabilities definitely improved at the higher PV by use of chalk-resistant pigments, but dirt collection definitely increased to poor appearance.

2. With 100 per cent Q viscosity linseed oil (air-bodied):

Durability perfect at 28.5 per cent PV and at 32 per cent PV. Durability practically perfect at 35 per cent PV and definitely superior to the paints at 35 per cent PV with 92 per cent alkali-refined linseed oil plus 8 per cent Q viscosity linseed oil, probably owing to improved weathering resistance of the 100 per cent Q viscosity oil.

3. With 100 per cent Z (22.7 poises) viscosity linseed oil (vacuum-bodied):

Perfect durability at 28.5 per cent PV. Practically perfect durability at

32 and 35 per cent PV. Slight cracking failure at 40 per cent PV.

4. With 100 per cent Z viscosity linseed oil (air-bodied):

Durability satisfactory at 28.5 per cent PV. Failure showing at 32.5 per cent PV and progressively worse through 35 per cent PV and 40 per cent PV.

5. With 100 per cent Z4 (63.4 poises) viscosity linseed oil (vacuum-bodied):

Durability satisfactory at 28.5 per cent PV. Slight failures at 32, 35, and 40 per cent PV. These failures start as loss of adhesion at the edges of the siding, curling away to expose bare wood.

6. With 100 per cent Z4 viscosity linseed oil (air-bodied):

Failures at all pigment volumes. Again these failures occur as loss of adhesion at the edges of the siding, curling away to expose bare wood.

7. With 100 per cent Z6 (148 poises) viscosity linseed oil (vacuum-bodied):

Failures at all pigment volumes (failures similar to 6).

8. With 100 per cent Z6 viscosity linseed oil (air-bodied):

Failures at all pigment volumes. Similar to the failures under 6 but more severe.

CORROSION

As in the past, today the most commonly used means of combating metallic corrosion is painting. New alloys, electroplating, surface film treatment, enamels, and other protective measures are being employed to protect base metals against corrosion, but paint still remains the most widely used means of extending the useful life of ferrous and non-ferrous metals and alloys.

Factors and forces operative in a corrosion cell are very complex and constitute a specialized study in themselves. Because of the complexity of corrosion study and the time which would necessarily be involved in giving even a brief explanation of corrosion fundamentals, the subject will be treated only as regards the mechanism by which paints function to combat corrosive forces and some of the factors connected with their use.

Paints prevent corrosion in three ways: (*a*) mechanical, (*b*) inhibitive formulation, and (*c*) galvanic protection. Paints, in all three mechanisms, act to guard against continued electrochemical corrosive attack, the most common force of corrosion.

The electrochemical process of corrosion is a condition under which a metal goes into solution in an ionic condition with the displacement of less electropositive ions from the solution. For example, when iron rusts, ferrous ions go into solution at some point or points and by so doing displace hydrogen from the solution. The reaction is as follows:

$$1.\ Fe \rightarrow Fe^{++} + 2e$$
$$H_2O \rightarrow H^+ + OH^-$$
$$2.\ 2H^+ + 2e \rightarrow H_2$$

This hydrogen escapes either as dissolved or gaseous hydrogen, leaving an excess of hydroxyl ion which renders the system alkaline. The driving force behind this reaction is associated with potential differences between point of solution and point of displacement.

This reaction may be followed visually by use of the familiar ferroxyl reagent. If a piece of steel is placed in a jelly containing a few drops of potassium ferricyanide and a few drops of phenolphthalein, after an hour or two blue areas appear where the ferrous ion from the metal has reacted with the ferricyanide. At other areas the excess of hydroxyl ion (OH^-) turns the phenolphthalein indicator pink. This initial step in rusting does not take place until water, coming through the paint film, reaches the metal.

Mechanical protection by means of paint relies upon isolation of the metal from the exposure environment and also upon the ability of the coating to insulate the anodic and cathodic areas from the necessary aqueous bridge between them.

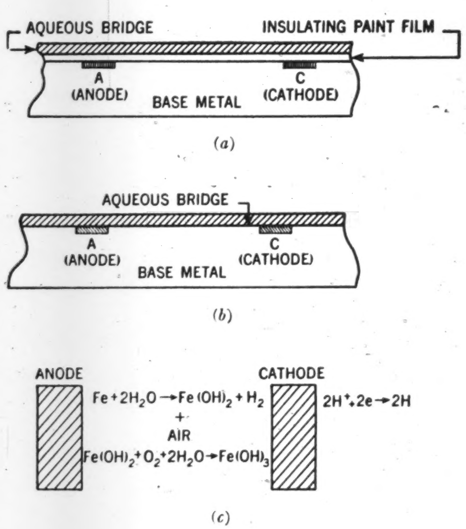

FIG. 7.

This is indicated in Fig. 7 (*a*). The paint film merely acts as a waterproof coating or electrical insulator between areas *A* and *C*. These areas must have a closed circuit in order to operate as a corrosion cell. It is a simple form of protection. There is, however, a major disadvantage to this type of paint protection. If the surface to be protected is not completely covered with a film that is free of pores, the corrosion process may be concentrated at these anodic areas, which usually constitute a small portion of the active area. This type of attack results in a pitting form of failure rather than a more generally distributed form of corrosion. Mechanical damage to the film after application has the same effect. The anodes and cathodes of a corrosion cell are not necessarily far apart and a small pore or scratch is sufficient to expose or create anodic and cathodic areas.

There are, however, distinct advantages to mechanical film protection, particularly in finish coats of especially prepared paints, which can be used in strong acid and alkali environments. Mechanically protective films are used with success only where there is assurance that complete coverage of surface is possible with minimum opportunity for accidental piercing of the film and where periodic inspection permits quick repair of broken films. It should be pointed out, however, that pore-free films are extremely difficult to obtain.

The second type of protective paints (those formulated with inhibitive pigments) are particularly useful because they are not as sensitive as the mechanical type of film towards small breaks or pores in the coating, and moisture impermeability does not have to be as complete to insure prolonged protection. This desirable feature is due to the passivating action of this type of paint.

When some metals are pre-exposed or remain in contact with certain reagents, their surfaces become passive or inactive towards environments in which they will normally corrode owing to the formation of self-healing protective films. Inhibitive pigments can also act as a source of passive reagent. These pigments depend upon the availability of limited amounts of moisture in order that the ions responsible for producing passivation may reach the metal surface.

Further explanation of corrosion phenomena and passivation may be made using iron as an example. Considering the base metal to be iron in Fig. 7 (*b*), the usual sequence of corrosion events would be as shown in Fig. 7 (*c*): (1) solution of iron at anode as ferrous iron; (2) formation of ferrous hydroxide; (3) oxidation of ferrous to ferric hydroxide.

The solubility of ferrous hydroxide in neutral solutions is greater than that of ferric hydroxide, allowing the ferrous material to diffuse some distance away from the point of origin before it precipitates out as ferric hydroxide because of its lower solubility. However, if the iron forms an adherent oxide film in intimate contact with the base metal, it stops further solution of the metal underlying it, thus passivating the metal. Protection in this case is established at the anode by preventing further solution of the metal; in other words, by oxidizing the ferrous ions at the point of solution, the insoluble ferric hydroxide covers the anode effectively. Such action might be considered analogous to the thin oxide film which forms on aluminum naturally and so effectively protects that metal without any apparent change in appearance. A similar type of film forms on steels of stainless variety. The more widely used ferrous metals do not have this inherent property, and protective measures must originate outside the metal. Effective passivation is provided primarily by the inclusion of inhibitive pigments such as red lead, zinc chromate, basic lead chromate, and others. When incorporated into paints, these pigments inhibit the process of corrosion by essentially functioning as oxidizing agents or by providing a mildly alkaline environment at the metal-paint film interface. One of the many illustrations that could be used to show arrested and active corrosion as described is the ferroxyl jelly test previously discussed. A clean iron nail placed in the jelly shows blue areas where the ferrous ion from the nail has reacted with the ferricyanide of the reagent.

Also the hydroxyl ion liberated from the water in areas has turned the phenolphthalein indicator from colorless to red. If now to a similar cell a few drops of any soluble chromate or dichromate such as the sodium or potassium salts is added in addition, no blue of ferrous ion or pink of hydroxyl appears. The solution potential of the metal to displace hydrogen is inhibited, and the nail will remain clean and bright for months.

In formulating a paint which has inhibitive properties, more is involved than merely including an active passivating agent. First, the reagent must go into solution in concentrations sufficient for effective protection. Second, the rate of such solubility must be adequate to insure protection as soon as moisture reaches the metal. Third, the rate of solution and effective concentration required must be slow enough to insure reasonably long-term protection to the base metal.

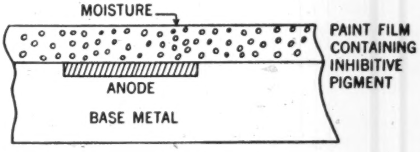

FIG. 8.

Assuming, for purpose of further discussion, that inhibitive pigments meeting the three mentioned requirements have been obtained, we may mention some problems associated with vehicle.

If we combine a vehicle of very high moisture impermeability with some inhibitive pigments, we may be defeating our own purpose. Referring to Fig. 8, let us consider the pigment to be held in a very impermeable vehicle. Moisture diffusing through the film must diffuse through the vehicle envelope about each pigment particle in order to dissolve the inhibitive ions of the pigment. The solution now carrying the inhibitive ions must re-diffuse away from the particle down to the metal interface if the ions are to become effective. It can be demonstrated that in some combinations of vehicle and pigment, moisture will diffuse down to the metal and cause corrosion before the necessary concentration level of inhibitive ions is reached by the solution. Thus an impermeable vehicle may make the reactive pigment inoperative for some time.

This explanation holds particularly for thin primer coats. Thick coats and multiple coats create some changes in the picture as the rate of moisture diffusion is retarded, but the possibility still exists of not making immediate and full use of the inhibitive ions as moisture passes down through the film to the metal.

A vehicle may be relatively permeable initially, yet the rate of pigment solution may be such as to permit a higher than necessary concentration of inhibitive ion with resultant early loss of effective film life.

An important factor connected with inhibitive pigments which warrants consideration is the chemical and physiochemical nature of environmental solutions to which an inhibitive paint is exposed. It is well known that paints that are entirely satisfactory for one environment may fail when used on the same type of base metal and structure in another environment. Some inhibitive pigments, because of their chemical reactivity, perform best in neutral or alkaline atmospheres, and are not suitable for use in industrial atmospheres because of the acid nature developed in solutions exposed to these conditions. A reagent capable of producing passivity in one environment does not always produce effective passivity in another environment. This failure is related to certain anodic or cathodic reactions sensitive to contact solution characteristics. For example, the pH of the solution may be such as to favor a reaction resulting in formation of compounds or precipitates other than those effective in preventing corrosion.

The effects of a change in contact solution or environment are reflected in initial and subsequent chemical reactivity and also in the rate of solubility of the pigment. Furthermore, the physical properties of the vehicle itself may alter with changes of exposure.

The major advantages of inhibitive type paint films may be considered as (*a*) their effectiveness in preventing corrosion even though minute breaks in the film are present that permit their effective use on structures which in practice are difficult to paint over with a completely pore-free film and (*b*) their effectiveness in paint films which are not completely impermeable to moisture. For maximum effectiveness, the exposure environment should be considered before formulating the paint.

A third and somewhat limited group of pigments offer electrochemical protection to the base metal but do not alter the surface of that metal. They do not necessarily attempt to keep moisture away from the base metal. Metallic pigments are electropositive to iron and fall in this group. Their action is similar to that afforded to steel which is covered by some metallic coatings. The zinc (on galvanized sheets) is sacrificial to the steel in that it dissolves or corrodes in preference to the ferrous metal, thus making the steel a cathode. Formulations based upon use of such pigments have effective life only so long as the protective pigment can go into solution. After solution of the metallic pigment is completed or halted, the remaining film offers only mechanical protection. Failure of such pigments rarely occurs simultaneously over the entire surface. It generally occurs first at isolated areas, and then the area of failure gradually extends until the whole surface ultimately fails. The environment in which this type of pigments is used must be such that the surface of the metallic particles does not become covered with an insoluble reaction product which would prevent continued solution of the sacrificing metal.

Chemical pretreatment methods for inducing passivity to metal surfaces before the application of organic coating are discussed in the section on "Surface Preparation."

SURFACE PREPARATION

The importance of surface preparation in extending the life of protective coatings cannot be overemphasized. In general, the useful life of any paint finish depends not only upon proper formulation of the protective paint but also upon the adherence of the film to the surface to which it is applied. Prolonged adherence depends largely upon the preparation of the surface before painting.

The importance of surface preparation of structural steel and its effect on the life of paint coatings in retarding corrosion has been stressed by Rassweiler, Diehlman (16), and others. They have pointed out that improving coatings by increasing the film strength, the impermeability to water and gases, the resistance to water disintegration, and the chemical resistance have not in themselves solved the problem.

The cause of failure of many protective coatings can usually be found in the lack of care which has been exercised in insuring a clean surface for painting. In the past it has been realized to some extent that oils, grease, and dirt generally would have a deleterious effect on paint adherence. It now is generally acknowledged that mill scale and rust can be equally detrimental to any satisfactory painting performance.

The aggregate of methods and materials for surface preparation of metals that have been exploited and described in the literature for the past ten years is quite impressive. However most of them are applicable only to small articles and are entirely impractical for the usual structural steel cleaning programs. Most methods of surface preparation can be placed in the following categories:

A. Removal of Foreign Matter

1. Mechanical
    (*a*) Wire brushing
    (*b*) Chipping
    (*c*) Sandblasting (dry and wet)
    (*d*) Shot blasting
    (*e*) Flame cleaning
2. Chemical
    (*a*) Alkali washes
    (*b*) Solvent degreasing
    (*c*) Pickling
    (*d*) Weathering

B. Chemical Pretreatments

1. Phosphatizing
    (*a*) Dilute phosphoric acid
    (*b*) Special commercial modifications, which convert the surface of the metal to a corrosion-inhibiting phosphate coating
    (*c*) Combinations with other inorganic acids
2. Chromatizing
3. Other chemical treatments

From a practical point of view, the whole question of steel surface preparation appears to fall logically into two main divisions; structural steel and pressed or sheet steel.

STRUCTURAL STEEL

Among the procedures of surface preparation for structural steel, weathering, followed by scraping and wire brushing, is widely practiced; sandblasting and pickling are used to a less extent. Their relative merit is unfortunately inversely proportional to their use in the field. Unless removal is complete, the practice of weathering to remove mill scale and other foreign matter is the least desirable of all the methods. Many tests have shown that partial removal of mill scale by weathering and wire brushing gives a very poor surface for painting. This is due in part to the hygroscopic nature of the residual rust and to the fact that this rust prevents intimate bonding of the paint vehicle to the base metal. Reasonably intact mill scale that has not rusted is a better surface for paint than scale which has been weathered. This fact has been appreciated to a great extent by the steel fabricators who have recently taken a more favorable attitude toward supplying steel that has received a shop paint coat before shipment and exposure. The other two methods of preparation, wet or dry sandblasting and pickling, give a completely clean surface and insure good adherence. While these methods are more costly initially, the increase in paint performance will in the long run result in a saving. Surfaces prepared by these methods should be painted almost immediately since rusting will quickly occur after complete cleaning. Flame cleaning, a more recent innovation, has found some acceptance.

Chemical treatments of many varieties have been developed for cleaning steel surfaces. In most instances these treatments are not applicable to large steel shapes, but it is reasonable to expect that a practical chemical process will be developed for large pieces since the effectiveness of chemical treatment has been demonstrated on smaller steel shapes.

The American Society for Testing Materials, The American Institute of Steel Construction, and The Iron and Steel Institute of England have conducted extensive investigations in the field of surface preparation and corrosion.

The need for surface preparation was emphasized by the last named in "Protective Painting of Structural Steel," a brochure based mainly on the experimental work of their Corrosion Committee's Subcommittee on Protective Coatings (42). The conclusions they advanced are supported by the results of workers in this country, and it is fair to state that the principles enunciated have gained general acceptance. Their conclusion and practical recommendations for painting are quoted below:

"Conclusion"

"1. Maximum durability is achieved by painting over a surface that has been completely freed from mill scale and rust.

"2. It is detrimental to allow a surface to rust again between the removal of the mill scale and rust and the application of the paint.

"3. Painting over a *complete* film of mill scale may give good durability in some cases.

"4. The life of paint applied to a surface from which mill scale and rust have been incompletely removed is materially reduced."

"Practical Recommendations"

"1. Obtain a good surface free from mill scale and rust. This is best done by pickling, sandblasting or shot-blasting.

"2. Paint the surface immediately after it has been prepared.

"3. Use a corrosion-inhibiting priming paint, such as red lead. Follow this with

Fig. 9.—Paint Failure Due to Lifting of Mill Scale from Steel Surface.

Painting scheme: Two coats of red lead; two coats of red oxide. Surface condition: intact mill scale. Exposure: Congella, South Africa; 5.8 years.

Courtesy of J. C. Hudson, D.Sc., Corrosion Committee, The Iron and Steel Inst., England.

Fig. 10.—Improved Paint Film Due to Removal of Mill Scale.

Paint scheme: Same as Fig. 9. Surface condition: Mill scale removed by pickling. Exposure: Same as Fig. 9.

Courtesy of J. C. Hudson, D.Sc., Corrosion Committee, The Iron and Steel Inst., England.

good weather-resisting finishing coats, such as oxide-of-iron or white-lead paints.

"4. Apply all paints under good conditions.

"If the painting of new iron and steel structures is carried out with due consideration to the principles outlined above, the saving in maintenance will more than repay the costs of surface preparation before painting."

Figure 9 illustrates the failure of paint from the lifting of mill scale, whereas Fig. 10 shows an improved paint film due to the effect of removing mill scale by pickling from steel before painting. Both panels received two priming coats of red lead and two finish coats of red oxide. They were exposed at Congella, South Africa, for 5.8 yr.

It is interesting to note the rate of corrosion of the steel of a bridge, as affected by two methods of preparation—wirebrushed and sandblasted—before the application of the priming coat. Figure 11 shows the corrosion condition after two years of exposure.

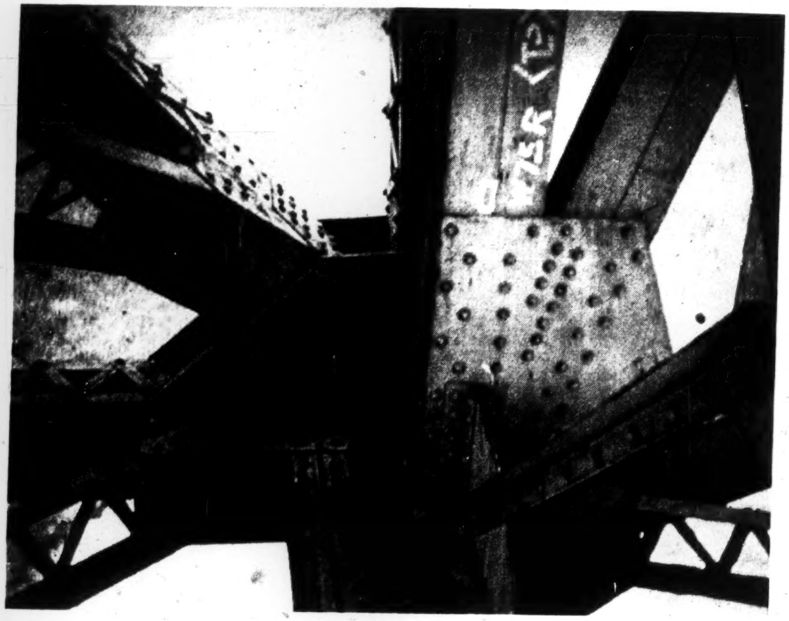
FIG. 11.—Shopcoat on Bridge, Two Years' Exposure Showing Steel Cleaned by Two Methods.
Left—Wirebrushed steel body rusted.
Right—Sandblasted steel in good condition.

PRESSED STEEL

Sheet steel which has been pressed into shapes such as automobile bodies and refrigerator shells must receive the same careful attention as structural steel.

Thorough preparation of the metal by removing rust, loose scale, oil, grease, and dirt is essential before the application of any protective paint. The treatments recommended usually have as their main objective the formation of a stable and less reactive coating combined with the base metal, a coating which not only retards corrosion but also provides greater adhesion of applied paint films. Any method of preparation which changes the surface characteristics of the metal is likely to have a positive effect on the adhesion and durability of a protective coating.

Darsey (15) classifies and describes some of the most widely used methods of metal preparation for sheet steel as follows:

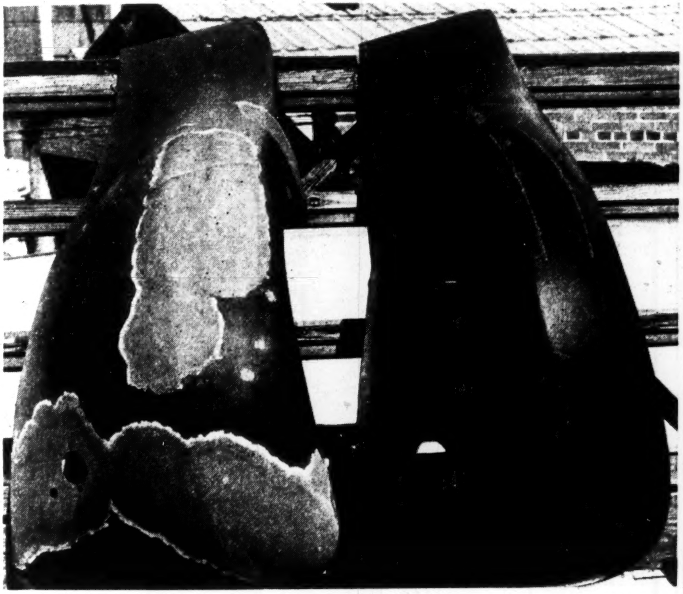

FIG. 12.—Effect of Surface Preparation of Paint Failure on Automobile Fenders.

Paint scheme: Two coats of black enamel.
Surface preparation: Left—solvent cleaned. Right—phosphate coated prior to application of paint.
Exposure: Six years in Detroit.

Courtesy of Parker Rust-Proof Co., Detroit, Mich.

Group I—Methods which clean without imparting any substantial change in the surface characteristics of the metal. The maximum results obtainable from their use are clean metallic surfaces. They do not provide for inhibiting corrosion or increasing the adhesion of applied paint coatings.

Group II—Methods which clean and chemically or mechanically change the surface characteristics of the metal. Representative are the use of acid pickling to remove rust and scale, and phosphoric acid cleaning solution (proprietary solution) to remove grease and oil and etch the metal surface. These etching and mechanical surface roughening methods increase adhesion of applied paint coatings but do not provide for inhibiting corrosion of the metal.

Group III—Methods which pre-clean the metal and convert its surface to a corrosion-inhibiting phosphate coating. Instead of increasing and activating the metal surface by etching or roughening to promote paint adhesion, these methods convert metal surfaces to a more stable and less reactive condition for the application of paint coatings.

The corrosivity of a metal is known to affect the protective life of applied paint coatings. Cold-rolled steel and zinc-coated panels which have been pretreated for painting by three different methods—solvent cleaning (trichloroethylene), phosphoric acid containing an organic solvent, and phosphate coating—exhibit different corrosion rates on outdoor exposure, humidity and salt-spray test.

A paint of given composition and

thickness applied to steel automobile fenders is shown (Fig. 12) to exhibit a marked difference in protection against corrosion after $6\frac{1}{2}$ yr. outdoor exposure, depending upon the method of surface preparation. One of the fenders was solvent cleaned and the other phosphate coated, both received the same dip primer and finish coat of black synthetic enamel. A diagonal scratch was made in the paint finish prior to exposure to determine the amount of corrosion creepage under the paint film from an abraded area. The results show that the same paint coatings afford much less protection when applied over solvent-cleaned steel than over phosphate-coated steel.

In Fig. 13 are shown results on a

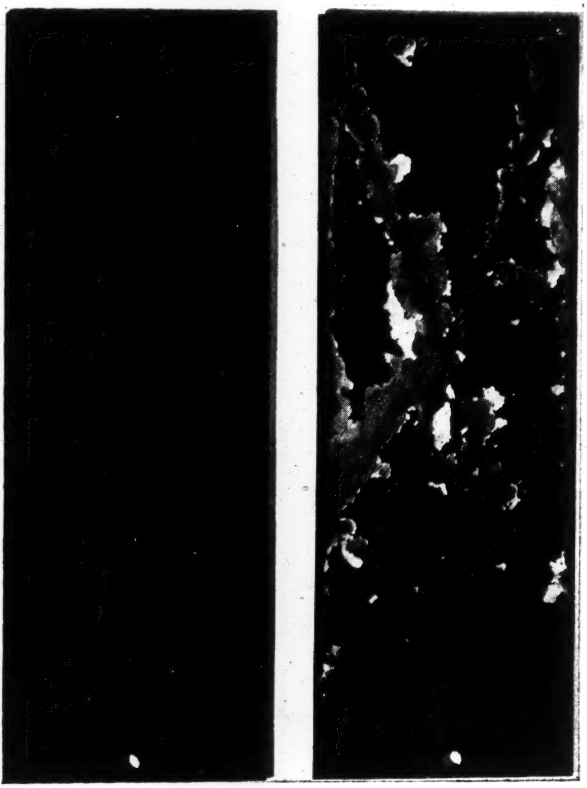

FIG. 13.—Effect of Surface Preparation of Paint Failure on Galvanized Panel.

Painting scheme: Same on both panels.
Surface preparation: Left—solvent cleaned. **Right—phosphate coated.**
Exposure condition: Six and **a half years in Detroit.**

Courtesy of Parker Rust-Proof Co., Detroit, Mich.

TABLE IV.—PAINT RETENTION AS EFFECTED BY METHOD OF SURFACE PREPARATION.

Primer—U. S. Army Specification 3-171.
Dipping Viscosity—24 sec. No. 4 Ford Cup at 80°F.
Dipped—Withdrawal rate 5 in. per min. at constant speed.
Weight of primer obtained by weights before and after painting.

	Primer, oz. per sq. ft.	Increase, per cent
Steel solvent cleaned	0.139	....
Steel phosphate coated[a]	0.177	27.3
Steel phosphate coated[b]	0.200	43.9
Steel phosphate coated[c]	0.242	74.0

[a] Phosphate coated by brush and roller application.
[b] Phosphate coated by spray application.
[c] Phosphate coated by immersion application.

galvanized panel, organic solvent cleaned, and a similar panel phosphate coated, each finished with the same composition paint and exposed outdoors for 6½ yr. Loss in adhesion of the paint is extensive on the solvent-cleaned panel.

The retention of paint on metal surfaces is an important factor where its application is by dipping or flow coating and the articles contain sharp edges. That phosphate coating of cold-rolled steel improves its paint retention characteristics when dipped and withdrawn at a uniform rate is shown in Table IV.

ENGINEERING ASPECTS OF COATINGS

The latest Webster's dictionary defines "engineering" as: "the arts and sciences by which the properties of matter and the source of power in nature are made useful to man in structures, machines and manufactured products." In view of the fact that they are an integral component as a material of construction and have specific functional properties in all engineering fields, protective organic coatings are definitely engineering materials.

It is true that almost up to a decade ago the engineer and the busy executive looked upon paints as a vague mixture of pigments and oils that gave him considerable trouble and annoyance and that offered at best only moderate protection. Dr. Speller, one of our outstanding engineers, as late as 1935 stressed the engineer's need for more information and a better understanding of the paints required to protect and perpetuate the metal structures which he designs and builds. In his book Corrosion (47) he states: "Too often the architect and the engineer are content to follow tradition in specifying materials and have been more or less indifferent towards the serviceable life rendered by the structures they design." However, during the past decade a great change has developed in the engineer's attitude toward paint.

During the past twenty or more years, a tremendous amount of fundamental technical and engineering data has been developed on the structural, physical, chemical, and functional properties of protective coatings for all engineering fields. These data are published in the various important technical publications.

It is impossible to discuss in one lecture or paper in detail all the engineering fields that use protective coatings. Therefore, only a few fields are presented, some of which are discussed in more detail than others.

ELECTRICAL INSULATION

The discovery of electricity and its application to human comfort and the necessities of life is one of the outstanding contributions of man. The average person in the home or in the factory does not appreciate the tremendous amount of research and engineering responsible for electrical development because its application is performed merely by pressing a button or pushing in a switch.

In the application of electricity or electric power, the protective coating industry has played a very important part in the past and will play a very important part in the future in the design and development of improved electrical applications. Without electrical insulation it is impossible to utilize electricity efficiently.

The generation of electricity at the power plant, its transportation through insulated wires to substations, its regulation through transformers, its flow to the industrial plant and the home, its transformation to light, heat, sound, and power through electrical appliances are obtained by the use of carefully designed electrical insulation of numerous types prepared from a wide variety of organic and inorganic raw materials formulated to meet mechanical, physical, chemical, and electrical requirements. As the research in electrical design brings to us discoveries and uses of electricity, engineering designs of improved insulation and new compounds with improved characteristics have to be developed to utilize those discoveries. Without improved insulation design, new electrical discoveries are not practical.

Among the important insulation products protected with or prepared from varnish and plastic coating compositions are the following:

Varnished Papers and Fabrics:

These materials, comprising what is commonly called flexible insulation, are used in practically every type of electrical apparatus in the form of tape, sheets, or punchings. The base materials commonly used are cotton cloth, silk, nylon, rayon, cellulose acetate, drills and ducks, kraft and rope condenser papers, saturating and super-calandered kraft papers, rag stock condenser papers, bond papers, vulcanized fiber, asbestos paper, and glass cloth.

Cotton cloth is furnished in two constructions, bias and straight-cut, and in a variety of counts and thicknesses. It is finished in a special manner by highly specialized finishes for electrical insulation purposes. This coated product is called varnished cambric (Fig. 14) and varnished cambric cable tape. The cable tape is used for power cables and specific types of small cables. The coatings are prepared from carefully designed varnishes to give low power factors, high insulation resistance, high dielectric strength, high tensile strength, high tearing strength, resistance to hot petrolatum and hot transformer oil, and finished to slide into place during cable wrapping. The physical, mechanical, chemical, and electrical properties are obtained from specific formulation of one or more varnishes applied in such a manner as to preserve the mechanical properties of the base cloth and finished for proper fabrication so as not to injure the electrical properties. Storage after production and packaging and storage before application are carefully designed for conservation of the original properties. Varnished cambric is prepared in yellow and black color. The preference is dependent upon the designer, but usually the yellow product is used where toughness and extreme oil resistance are desired. The

FIG. 14.—Varnished Cambric Used as Insulating Barrier Between Core and Low Voltage Winding of a Power Transformer.

Courtesy of National Varnish Products Corp.

black fabric is used in applications where high dielectric strength, high insulation resistance, and low power factor under adverse conditions are desired. The straight-cut cambric is used under normal applications whereas the bias-cut material is used over irregular surfaces and for cable splicing. For cable splicing the electrical properties must be preserved. In order to do this, freshly coated material is treated under oil and kept completely immersed in oil until application.

Coated silk (Fig. 15), nylon, rayon and cellulose acetate are used where high dielectric strength is desired and space is of prime importance. Low acid value and pH values between 5 and 7 are desired in these coated fabrics to prevent corrosion of fine conductors. These products are used as insulation in magnetos, small motors, small coils, small transformers, generators, and small electrical assemblies. Silk is used in some larger units over irregular surfaces where space is a factor and bias cloth is too thick. The resiliency of coated silk enables it to withstand this abuse.

Coated drills and ducks are used for phase and slot insulation and in heavy-duty equipment, such as mining equipment and large electromagnets, where mechanical properties are desired.

Coated kraft, rope, and rag condenser papers, and thin bond papers are used in the manufacture of coils, magnetos, condensers, and as punchings in layers in assembled electrical appliances. Coated saturates and super-calandered kraft papers are used in condensers, outside of coils, and as punching laminates in auxiliary apparatus. Coated vulcanized fiber is used as an armature slot insulation in formed resistors and in punchings in laminations in a good many electrical appliances. Uncoated vulcanized fiber and heavy tough slot papers are duplexed to varnished cambric and

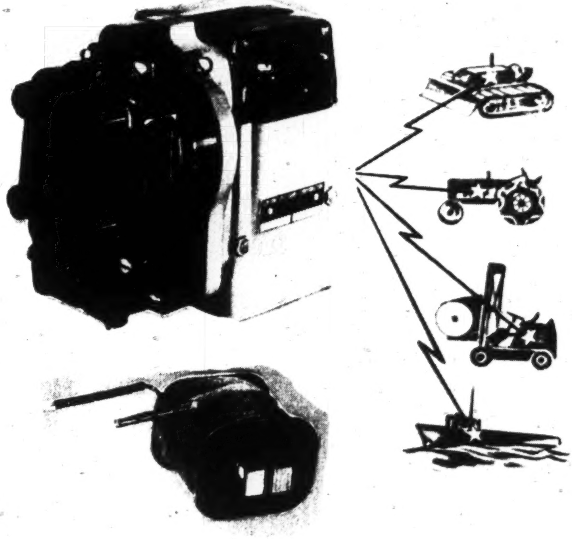

FIG. 15. Varnished Silk Used in High Tension Coils.

Courtesy of National Varnish Products Corp used as motor slot insulation. The papers give strong mechanical properties whereas the varnished cambrics possess good electrical properties.

Coated glass cloth is used in motor slot insulation, coils, transformers, in electrical equipment which operates at elevated temperatures, and as punchings for laminated assemblies.

The foregoing products are coated with varnishes prepared from vegetable oils, all types of resins, asphalts, blown petroleum pitches, waxes, heavy oils, greases, metallic driers, and many other varnish raw materials. Originally the coatings used were made primarily from linseed oil. Later, improvements were made with the addition of other oils such as tung (China wood), oil, dehydrated castor oil, soybean oil, cashew nut shell liquid. The oil lengths are exceptionally long for yellows and short to medium for the black cambrics. The varnishes predominate in tung (China wood) oil. Semi-drying oils and greases are used to obtain desirable softer physical properties. Nonheat-reactive 100 per cent phenolic resins have been used to decrease the oil length to produce improved heat-resistant varnishes. The major defects of varnish coatings in electrical insulation have been the continued degradation of the films under service strains which changes the electrical characteristics by producing an increase in power factor and a decrease in insulation resistance. These changes take place under adverse conditions such as operation under high humidities, high heat caused by overload, or location of cables in heated areas. Newer types of organosilicone resins show great promise towards high-temperature resistant insulating varnishes. The modern trend in coating development is stabilization under high humidities and high heat. Similar research is being conducted on the purification and stabilization of the uncoated base. Power losses by instability are considerable and can be decreased to a great extent by further research.

The development of organosilicone-coated glass cloth has opened a field in electric design of smaller-size equipment for equal power output. An illustration is the production of a 10-hp. motor occupying the space of that of a conventional 3-hp. motor. Aside from a space factor, organosilicone-insulated motors can be operated at higher temperatures and will take overloads without serious damage. The use of tafflon tapes is also an advancement in a stability product. The industry needs engineering design and efficiency to produce these organosilicone resins, glass cloth, and tafflon resins at a cost to be utilized economically in volume electrical insulation products.

Insulated Magnet Wire:

Magnet wire is prepared copper wire ranging in sizes from 0.0031 to 0.1285 in. (American Wire Gages Nos. 40 to 8), insulated with varnish enamels, covered or not covered with silk, acetate, nylon, cotton, or glass braid. It is used in the manufacture of an extensive variety of coils, transformers, relays, small motors, chokes for power supply in conjunction with rectifiers, some types of telephone cables, and in many cases where magnetism is required. Magnet wire is manufactured in circular, square and rectangular designs. The latter two types are used when it is desired to put a larger number of wires within a smaller space. The properties desired in insulating coatings are hardness, toughness, flexibility, continuity of film, stretch, moisture resistance, solvent resistance, high dielectric strength, oil resistance, heat endurance so as to produce a thin film without pinholes, resistance to insulating varnish solvents, enough toughness to withstand high-speed winding in gang coils, flexible enough to withstand stretch during tight winding and to withstand expansion and contraction caused by heating and cooling in the coil during operation, and high enough in dielectric strength to prevent a short circuit during the passing of the electric current. Wire enamels are prepared from vegetable oils and natural resin combinations, vegetable oils and phenolic resin combinations, adipic acid alkyd resin blended with alcohol-substituted urea-formaldehyde resins, polyvinyl formals, and polyvinyl formal blends with both alkyds and phenolics.

Insulating Varnishes:

Widely used materials such as cotton, paper, wood, and asbestos are excellent insulators when dry, but they absorb moisture and gases and in time would become conductors if not protected by a coating of insulating varnishes. These varnishes are applied to practically all types of electrical windings and assemblies to prevent electrical deterioration caused by moisture, conductive particles, dirt, heat, vapors, acids, oils, and they act as a binding medium forming a solid unit and becoming a part of the apparatus. These varnishes are prepared as baking, air-drying, and for

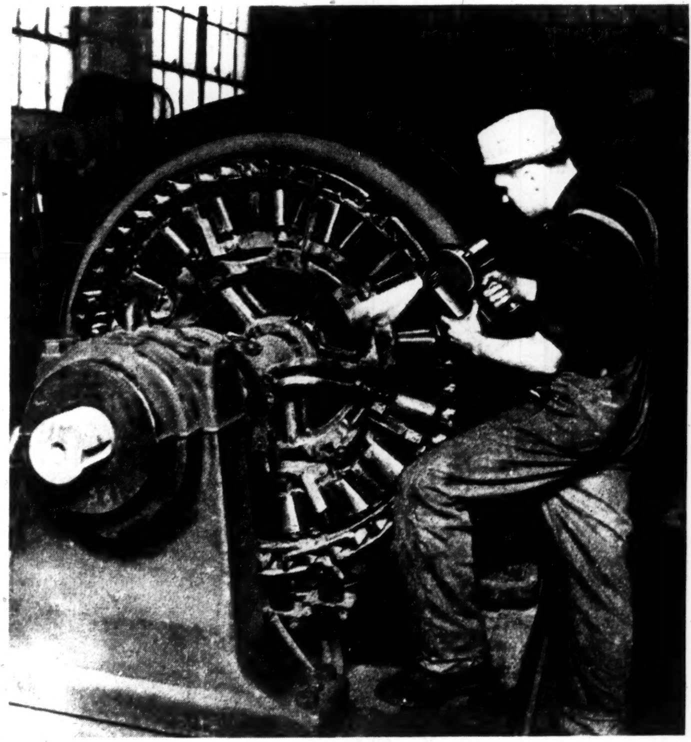

FIG. 16.—Motor Generator Sprayed with Air-Drying Varnish.

Courtesy of Irvington Varnish and Insulator Co finishing products, in both yellow and black color. The baking material is preferred to the air-drying because of its greater durability and flexibility and is less subject to atmospheric change. The black baking varnish finds wider application because of its higher dielectric strength both wet and dry, greater flexibility, and greater moisture resistance. The clear baking varnishes are preferred in many cases because of their greater oil-proofness, mechanical strength, and clear color for detection of defects. The air-drying varnishes, soluble in petroleum hydrocarbon, are prepared in two types: one drying by evaporation of solvent alone and generally not resistant to transformer and lubricating oils; the other drying by the evaporation of solvent plus oxidation and other normal chemical and physical changes. Air-drying varnishes are used where baking facilities are not obtainable and low-cost operation is necessary. See Figs. 16 and 17.

Finishing varnishes are alcohol-soluble compositions of extreme oil resistance, which are used alone or over other insulation varnishes where windings are to be washed frequently with gasoline and where lubricating oil and transformer oil are encountered.

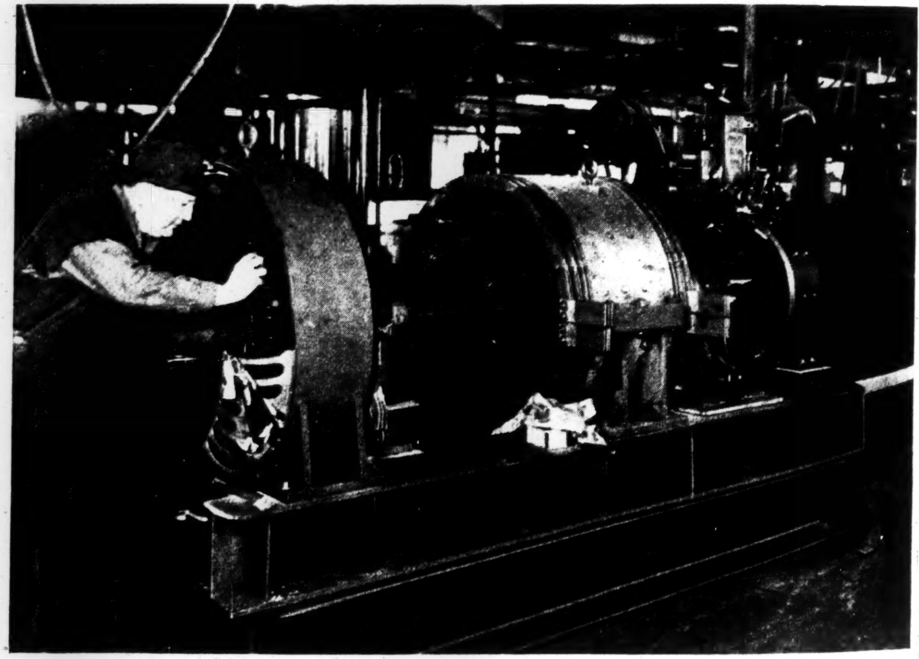

FIG. 17.—Coil in Motor Stator Receiving a Coat of Black Air-Drying Varnish.

Courtesy of Irvington Varnish and Insulator Co.

Primary among the problems for consideration in the use of organic insulating coatings are moisture and water resistance. The figures in the following table compare the percentage of water absorption of uncoated insulation and the same insulating material coated with an organic resinous film.

The test was conducted by immersion for 18 hr. at 70 F.

PERCENTAGE OF WATER ABSORPTION OF COATED *VERSUS* UNCOATED INSULATING MATERIALS.

Material	Uncoated	Coated 1¼ mils each side
Rayon	50	5.0
Cotton	30	5.0
Fiber glass	15	1.0

It can be seen from the above that coated products are far superior.

Considering the effect of moisture on the dielectric strength, we again find the coated products to be superior. The following table shows the dielectric strength after 18 hr. of immersion in water at 70 F.:

DIELECTRIC STRENGTH OF COATED *VERSUS* UNCOATED MATERIALS AFTER WATER IMMERSION.

Fabric	Volts per mil on Coated Product 1¼ mils each side	Volts per mil on Uncoated Product
Rayon	750	Approx. 50
Cotton	450	Approx. 50
Fiber glass	380	Approx. 50
Nylon	900	Approx. 50

NOTE.—The approximate figures are due to difficulty in readings because of evaporation during testing.

In exposure to high humidity, it is shown that organic coatings give improved properties over uncoated insulation. The following table shows the dielectric strength at 75 F. after exposure to relative humidity of 96 per cent for 96 hr.:

DIELECTRIC STRENGTH OF COATED *VERSUS* UNCOATED MATERIALS AFTER EXPOSURE TO HIGH HUMIDITY.

Fabric	Volts per mil on Coated Product 1¼ mils each side	Volts per mil on Uncoated Product
Rayon	350	Approx. 50
Cotton	510	Approx. 50
Fiber glass	310	Approx. 50
Nylon	270	Approx. 50

Here it is obvious why coatings are used under humid conditions. The reason for the apparent error in giving higher dielectric strength to cotton than to fiber glass is due in this case to a greater penetration of the coating into the fibers of the cotton whereas the glass left a greater quantity of voids. *All the above figures are merely comparative with one type of coating and do not represent the best coating for these purposes.*

The effect of moisture on power factor on coated and uncoated fabrics is shown in the table below. Coated samples were exposed 24 hr. at 65 per cent relative humidity. Uncoated samples were exposed 1 hr. at 65 per cent relative humidity. Power factor measurements on samples were made at 212 F.; samples in the column marked "before" were previously conditioned at 230 F. Figures are for before and after exposure.

POWER FACTOR OF COATED *VERSUS* UNCOATED MATERIALS AFTER EXPOSURE TO 65 PER CENT RELATIVE HUMIDITY.

Fabric	Power Factor on Coated Fabric, per cent		Power Factor on Uncoated Fabric, per cent	
	Before	After	Before	After
Rayon	5 to 8	30	Approx. 5	Over 50
Cotton	5 to 8	15	Approx. 2	Over 50
Fiber glass	3 to 5	10 to 15	Approx. 2	Over 50
Nylon	3 to 5	15 to 20	Approx. 2	Over 50

Here again we find that under humid conditions the uncoated fabrics are inferior and give such high power factors and power losses as to make operations uneconomical, especially when alternating current is used. The effect of dielectric loss due to high power factor on current-carrying capacity of a cable is of small consequence when the voltage is low, of the order of 600 v. or less; but when the voltage is of the order of 13,000 v., then the loss factor becomes of great importance. High power factor will induce heating within the insulation with a corresponding increase in line loss and a decrease in the current-carrying capacity of the cable.

End-use requirements of insulating varnishes are resistance to transformer and lubricating oils, heat endurance, flexibility, bonding strength, adhesion, acid resistance, penetration, uniform evaporation rate, moisture resistance, compact film build-up, good heat conductivity, low susceptibility to temperature change, abrasion resistance, low solvent retention, homogeneous compact film on drying, no deleterious effect on enameled wire toughness, stability in temperature changes, ozone resistance, high dielectric strength (dry and after subjection to high humidities), high insulation resistance dry and under adverse conditions, low power factor and arc resistance, stability of liquid varnish on aging and after repeated thinnings. No one type of varnish meets all these requirements so that it is necessary to make a considerable number of varnishes.

The conventional types of insulating varnishes are made from drying oils, resins, asphalts, pitches, metallic driers, and solvents selected to meet requirements based upon the properties of each of the individual constituents.

Short and medium oil length baking insulating varnishes are used on armatures and field coils before assembly, on wound armatures and stators, small rigid and semi-rigid coils, small high-speed fans, dental motors, small air-cooled transformers, on apparatus where bonding strength is necessary, and for applications where acid resistance is required. Long oil baking varnishes are used on form wound coils (Fig. 18) of all types, oil-cooled transformers, coils and assembly units, railway industrial armatures and stator coils, railway mining and heavy-duty apparatus, and apparatus that is subjected to severe and sustained vibration. Short oil air drying varnishes are used on conduit boxes, switches, signal boxes, storage battery trays, motor frames, castings, and metallic surfaces.

The more recent trend in improved insulating baking varnishes, particularly for deep impregnations, has been what is termed "deep dry" varnishes. These varnishes are designed to cure mostly by polymerization and condensation to a solid which is completely dry throughout the entire mass. Varnishes of this type are prepared from cashew nut shell oil

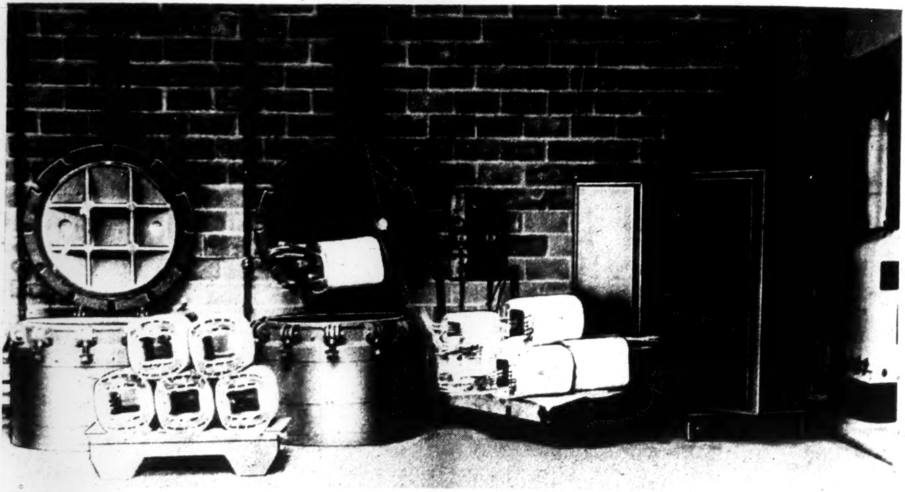

FIG. 18.—Transformer Coils Being Lowered into a Vacuum Impregnating Tank for Treatment with Baking Insulating Varnish.

Courtesy of Irvington Varnish and Insulator Co.

condensation products, Fosterite (Westinghouse patented compounds), heat-reactive 100 per cent phenolic resin and blown oil combinations, synthetically prepared catalytic oils with and without heat-reactive resins, and high molecular weight elastomers. These combinations are prepared with and without solvents.

The most recent development in insulating varnishes has been the organosilicones, which are used in all glass-insulated apparatus. From the properties of the organosilicone resins, electrical apparatus thus treated can operate at higher temperatures, permit overloads without damage, and allow a considerable saving in space in newly designed equipment. By reducing motor failures caused by excessive moisture plus heat from overloading, organosilicone insulation can cut maintenance cost to a minimum.

It is interesting to note in Fig. 19 the different methods of insulations employed in the compass coil—Navy K2. The coils were used on all ships for the purpose of rectifying compass deviations by the ships' degassing equipment.

The effect of moisture on the electrical properties of varnished and unvarnished coil windings is shown in the following test. Each coil had two windings to enable measurement of insulation resistance and dielectric breakdown between windings. The coatings were made with an interior drying baking varnish (♁) and the coils were preheated 1 hr. at 270 F., dipped in varnish for one hour, and baked 4 hr. at 270 F. The second coat was dipped one minute, then baked an additional 4 hr. at 270 F. The results of this test are given in Table V.

We see that in this case unvarnished coils would be useless in the presence of water even though the copper wire had a glass or cotton covering.

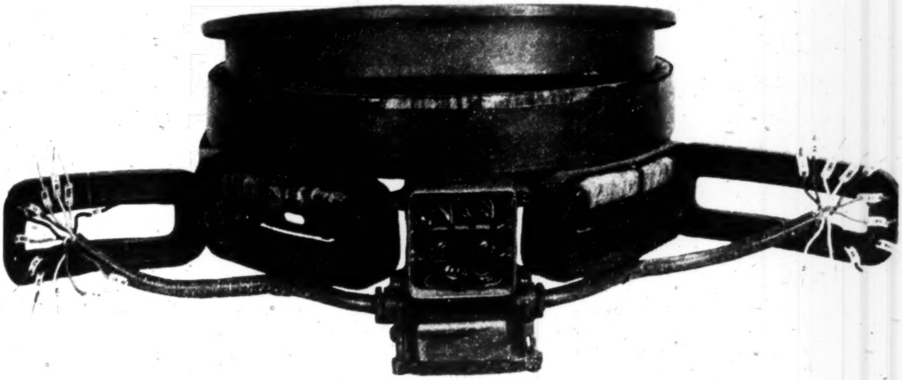

FIG. 19.—Compass Coil—Navy K2.

Fiberglas insulation on all coils which are then coated with varnish.
All leads insulated with varnished rayon tubing.
Terminal box:
  Varnished Fiberglas insulation.
  Terminals insulated with extruded vinyl chloride.
  Block made of laminated phenolic—linen and canvas bases used.
  Cable lines coated with extruded vinyl chloride; colors used according to code for identification.
  Coating of paint on metal.

Courtesy of National Varnish Products Corp.

Sleeving and Tubing:

The insulation product known as sleeving or tubing is a hollow tube ranging in sizes from 0.024 to 2.0 in. in inside diameter and supplied in standard electrical code colors. It is manufactured as an extruded tube or a coated braid. The braid may be cotton, rayon, nylon, silk, or glass-coated with nitrocellulose, ethylcellulose, cellulose acetate, organosilicone, cellulose acetobutyrate, oleoresinous varnishes, or chlorinated rubber and supplied in standard products of saturated sleeving with no dielectric requirement, sleeving of 1500 to 2500 v., and tubing of 4000 to 7000 v. The braid offers mechanical strength, and the coating supplies the physical, chemical, and electrical characteristics. The extruded tubing may be prepared from rubber, synthetic rubber, neoprene, cellulose acetate, styrol, methacrylates, acetobutyrate, polyethylene, and vinyl compositions, all designed to meet physical, mechanical, chemical, and electrical characteristics of exacting specifications. The vinyl copolymers and polyvinyl chloride formulations have found wide application and are supplied from a rigid tube to an exceptionally flexible composition withstanding temperatures as low as −60 to −70 F. Transformer leads are specific types of tubing prepared for exceptionally high dielectric strength and are prepared from several wraps of varnished cambric covered with a cotton or rayon braid and saturated with insulating varnish to hold the roundness. Uncoated cotton, rayon, glass, and asbestos sleeving are also used by slipping over wire leads and the entire unit dipped in insulating varnishes, thus obtaining the electrical properties from the impregnating operation. Tubings and sleevings are formulated to have high dielectric strength, high insulation resistance, heat endurance, flexibility, nonfraying, resistance to hot compounds, oil-proofness, resistance to acids, alkalies, waxes, alcohol, oxidation, moisture, abrasion, shock, and fungus, permanence of color at high temperature, low-temperature flexibility, and low power factor at high frequencies.

TABLE V.—EFFECT OF MOISTURE ON ELECTRICAL PROPERTIES OF VARNISHED AND UNVARNISHED COIL WINDINGS.

Coil	Type of Insulation	Varnish Treatment of Coil	Insulation Resistance Between Windings at 75 F.	Dielectric Between Winding, v.	Insulation Resistance After soaking in Water $\frac{1}{4}$ hr., megohms	Dielectric After Soaking in Water 20 min., v.
No. 1	Glass-covered wire	None	Infinity	900	....	...
No. 2		None	Infinity	....	0	0
No. 3		Two Coats (a)	Infinity	1280	....	...
No. 4		Two Coats (a)	Infinity	....	1.7	1200
No. 5	Cotton-covered wire	None	Infinity	820	....	...
No. 6		None	Infinity	....	0	0
No. 7		Two Coats (a)	Infinity	1600	....	...
No. 8		Two Coats (a)	Infinity	....	1.0	1100

Tubings and sleevings are used as insulation on lead wires for all types of electrical apparatus, connecting component parts on switchboards, assemblies using short sections of wires, in the base of radio tubes, for meters, automobile harness, airplane harness, watertight and moisture-tight switch boxes, over wire assemblies, blown over conduit, motor and transformer terminal leads, connections in radio receivers and transmitters, electronic and magnetic control devices, and over solderless terminal lugs and splice connectors.

Wires and Cables:

Cables may be divided into three main groups:

1. Those for generating stations and substations where varnished cambric cables are usually used.
2. Those for transmission and distribution feeders where oil-impregnated paper cables are usually used.
3. Those for secondary systems where natural rubber and synthetic cables are used.

I. Varnished Cambric Cables.—Varnished cambric cables are used in installations from 1 to 28 kv. They are subdivided in three main classes:

(*a*) Single conductor used on installations from 1 to 28 kv.
(*b*) Multi-conductor nonbelted used on installations from 1 to 28 kv.
(*c*) Multi-conductor belted used on installations from 1 to 17 kv.

Single-conductor cables are the most prevalent, in that they are practical in making installations. The cables are prepared with and without a lead sheath and both shielded and nonshielded. Lead-covered cables are used where extreme moisture is present. The cables are shielded when installed for operation above 8 kv. Varnished cambric cables are more moisture resistant than oil-impregnated paper cables and, therefore, can be used in many places without a lead sheath such as control wires, street lighting wires, and where space is a factor, in that joints can be made without the use of balky potheads. Varnished cambric cables are also used on vertical risers such as tall buildings as there is no danger of oil migration to harm the insulation. Varnished cambric cables not lead-covered are supplied with a cotton braid asphalt impregnated, asbestos braiding impregnated with flame-retarding compounds, vinyl resin jacketed, rubber jacketed, and neoprene jacketed, each designed for a specific purpose or to meet exacting specifications. Aside from the layer cables, there are numerous small wire installations, especially in the fire-retardant groupings such as 600-v. applications of switchboard wire, control wire, power cables, boiler room wire, shipboard wire, and resistance wires which are composed of asbestos fillers, varnish cambric, and asbestos braids impregnated with flame-retardant compounds such as chlorinated paraffin and produced in the NEMA code colors (**38**).

II. Oil-Impregnated Paper Cables.—Best electrical insulation is afforded when the cables are protected from water or its vapor. This protection is obtained by the use of a lead sheath extruded over the paper cable. These oil-impregnated paper cables are subdivided into four types:

A. There are solid paper cables which are further subdivided into single conductor cables, both shielded and nonshielded, used in installations from 1 to 69 kv.; multi-conductor belted cables used in installations from 1 to 15 kv.; and multi-conductor shielded cables used in installations from 9 to 46 kv. These cables are spliced together with varnished cambric bias tapes and the splices are further protected by running through metal boxes called potheads, which are filled with compounds to insure protection around the joint. For cables of low voltage, medium-voltage single-conductor and three-conductor cables, air-blown conductor cables, where ease of removal is necessary, refined petroleum jelly is used. For higher voltages used under pressure at the joint, insulating petroleum oil is used in the pothead provided with a suitable reservoir. Several special pothead compounds and joint compounds are also used. They are combinations of blown asphalts and synthetic resins, combinations of synthesized oils and resins, and combinations of hardwood pitch and synthetic resins.

B. Oil-Filled Impregnated Paper Cables: The basic principle of these oil-filled cables is that all space within an insulated cable is kept completely filled with gas-free and moisture-free oil, and means are provided for free movement of the oil under heating and cooling cycles. The maintenance of treated oil within all parts of the cable at all times prevents any ionization in voids and, therefore, prevents electrical failure.

These cables are subdivided into single-conductor cables for operation between 15 and 230 kv. and three-conductor shielded cables for operation between 15 and 80 kv. These cables are the most expensive system of cable installation. The single-conductor cables are constructed with a hollow core or with a fluted lead sheath for free oil movement. The three-conductor cables are provided with channels in the three interstices for oil movement.

C. Gas-Filled Paper-Impregnated Cables: Gas-filled cable is a cable wherein a positive pressure is maintained with a dry inert gas, normally nitrogen. These cables are divided further into three classifications: (*a*) low-pressure cables used for installations between 9 and 40 kv. and operating under 15-lb. pressure; (*b*) medium-pressure cables used for installations between 45 and 60 kv. and operating under 45-lb. pressure; and (*c*) high-pressure cable used for installations between 69 and 138 kv. and operating under 200-lb. pressure. Varnished cambric bias tapes are used for the cable splices and for the higher voltages low-power factor oil immersed.

D. High-Pressure Pipe Oil Impregnated Cables: These cables, as the name implies, are installed in a steel pipe somewhat larger than the cables and subjected to 200-lb. pressure by the surrounding medium. These cables are normally single-conductor, and the pressure medium may be either oil or inert gas. High-pressure pipe systems with internal pressure require corrosion protection to avoid leaks. Where soil conditions are corrosive, particular attention has to be paid to complete coverage of the pipe with a protective coating. The use of electronic heating of the new types of phenolic coatings for pipe protection was used very successfully during the war and will aid greatly in these corrosive problems.

III. Natural Rubber and Synthetics.—Rubber-insulated wires and cables supplemented by organic protective coatings are considered the backbone of the insulation industry. Both natural and synthetic rubbers are compounded to meet the performance of the electrical codes. The most widely used rubber compounds are those containing 30 per cent rubber. They are suitable for building wires and other insulations up to 2000 v. protection. The highest grade of rubber insulation is known as ozone-resistant rubber, which can be used on installations of 2000 to 27,000 v. capacity. The rubber compositions containing rubber, carbon black, zinc oxide, fillers, vulcanizing chemicals, antioxidants, etc., are compounded on rubber mills and applied to the wire by both extrusion process and from calandered rubber tapes. The rubber tapes are applied to wires by specialized apparatus and vulcanized with and without a jacket. The jacket, which may be highly polished tin foil or lead, is stripped from the wire after vulcanization. After application of the rubber to the wire it is vulcanized, either immediately following the application of the rubber compounds to the wire or by a second operation, depending upon the compound and the size of the wire insulated. In addition to these processes of application, rubber is also coated on wire by rubber latex solutions, running the wire through these latex solutions and heat curing between dips. These rubber-insulated wires may be used with or without a covering. The common coverings are cotton, rayon and asbestos braids, thiokol, vinyl, and neoprene jackets. The braided coverings are protected by compound impregnation and various lacquer coatings. The impregnating compounds are asphaltic combinations, asphalt wax combinations, wax resin compositions, flameproofing compounds, and chlorinated naphthalene compounds. The lacquer coatings are prepared from nitrocellulose, cellulose acetate, cellulose acetate butyrate, cellulose acetate propionate, ethylcellulose, chlorinated rubber, and vinyl compounds. The plasticizers may or may not contain flame-resistant ingredients such as tributyl phosphate, triphenyl phosphate, chlorinated diphenyls, and chlorinated paraffins. These coatings protect the rubber from oil and oxidations. All these braid saturants or coatings are supplied in the NEMA code standards.

Synthetic insulated wire coatings are of both the thermosetting and thermoplastic types. Among the compounds used are buna S, buna N, neoprene, nylon thiokol, polyvinyl compounds, and polyethylene. The polyvinyl compounds are the most widely used. Their main application is for low-frequency operation in low-voltage power and communication cables where circuit voltages do not exceed 600 v. alternating current. The standard vinyl compounds are not recommended for continuous operation above 140 F. but there are special compounds which have been developed to withstand 185 F. continuous operation. The physical properties of the vinyls compare favorably with the best rubbers. Polyethylene insulated wire is used for high-frequency application. It has a high degree of toughness and stability, not being affected by sunlight or ozone. It also has very low moisture absorption.

Thiokol, nylon, and neoprene have found extensive use as jacket material for insulated wires because of good flexibility, toughness, resistance to abrasion, and acid-, alkali-, salt-, and moisture-resistance.

The importance of protective organic coatings in electrical insulation has placed their position high in the rank of engineering materials.

FIRE-RETARDANT PAINTS

There are few things so terrible as fire at sea. Ships, which have fuel tanks girdling their hulls, can be turned into floating infernos in an instant. The danger of fire is especially great on naval ships during action. Enemy shells and bombs ripping through their decks cause explosions which spread destructive fires in short time, sometimes propagated by heavy paint films.

Paint films are highly inflammable and evolve carbon monoxide and other gases when heated in sufficient quantity to be a poison or an explosive hazard under certain conditions. The hazard of thick films of paint resulting from forty or fifty coats on interiors of ships was clearly demonstrated during the war. A tremendous amount of heat was generated in a compartment in which an explosion occurred. The adjacent bulkheads and decks became heated, and sometimes when the painted metal was raised over 1600 F., a fire occurred, generally starting at a blister in which combustible gases were heated to the flash point. In place of the heat being dissipated over a large area, the burning paint melted and, sliding to the deck, concentrated the generated heat and sometimes transmitted it to another compartment.

As a result of this hazard, all ships started scraping down the interior painted bulkheads and decks to the bare metal in the summer of 1942. The dingy appearance of the interior when not painted is hard to imagine; however, the effect on lighting and personnel is obvious. It was an arduous task to scrape the paint to bare metal, especially on new ships delivered to the fleet because it was a thin tough coating which would not crack off like twenty or thirty coats of completely oxidized film.

The Bureau of Ships authorized work on a fire-retardant interior paint at the Philadelphia Navy Yard to formulate a product with the following attributes:

(*a*) Applied over a primed steel bulkhead and exposed to 1900 F. on the reverse side, the paint film should not catch fire.
(*b*) Should not emit toxic fumes when subjected to flame.
(*c*) No toxic gases when applied by spraying.
(*d*) Hide zinc chromate primer in one thin coat.
(*e*) Mildew resistant and rodent repelling.
(*f*) Not brittle enough to be a hazard when shattered off the bulkhead by concussion.
(*g*) Not rust exciting.

As the problem developed it became evident that coordinated research work would be needed and various phases of the problem were carried on at the Mare Island, Norfolk, and Puget Sound Navy Yards, with supplementary phases handled by the National Defense Research Council, the National Public Health Service, a number of commercial laboratories, and three of the local clubs of the Federation of Paint and Varnish Production Clubs. The Norfolk and Mare Island Yards made production batches to determine the manufacturing and application properties of some of the formulas, and service tests of the finished products were conducted at the Naval Fire-Fighting Schools at Norfolk and Boston. As a result of this interrelated research work, Fire Retardant Inside Semi-Gloss White Paint No. 27 was developed which depended upon the following attributes for its fire retardation:

(*a*) A very thin layer of paint of less than one mil is applied since it has high hiding power.
(*b*) The organic binder has been reduced to a minimum.
(*c*) As the dried film is heated to the flash point, a pigment therein decomposes which perforates the film and thus prevents the accumulation of combustible gases in the blisters formed.

The fire fighting in adjacent compartments painted with this paint is facilitated because the heat radiated is less than that radiated from a bare bulkhead. This paint has exhibited excellent service since the specification was issued on November 7, 1942.

FOOD AND CHEMICAL CONTAINERS, METAL

One of the most important contributions made by protective coatings toward making our everyday lives safer and more healthful is the protection of food cans, kits, and drums in which many of our daily foods are shipped and stored.

Everyone has noticed, upon opening a can of fruit or vegetables, the golden yellow sanitary lining that has been applied to the interior of the can. This golden lining is a product of the paint industry. It is odorless, tasteless, and nontoxic and is applied to the interior of the can for your protection against contamination of the food by the metal body of the container. Many canned foods contain ingredients that are acidic in nature which, upon long storage, would react with the tin. Alkaline vegetables and meats which liberate sulfur during processing would turn black in unlined containers. Thus the paint industry contributes to the daily health of many millions of people. Larger packages, such as 5-gal. kits containing beverage syrups and 55-gal. drums for shipping and storing salad oils, vegetable shortenings, lard, oleic acid, etc., are all protected by sanitary linings.

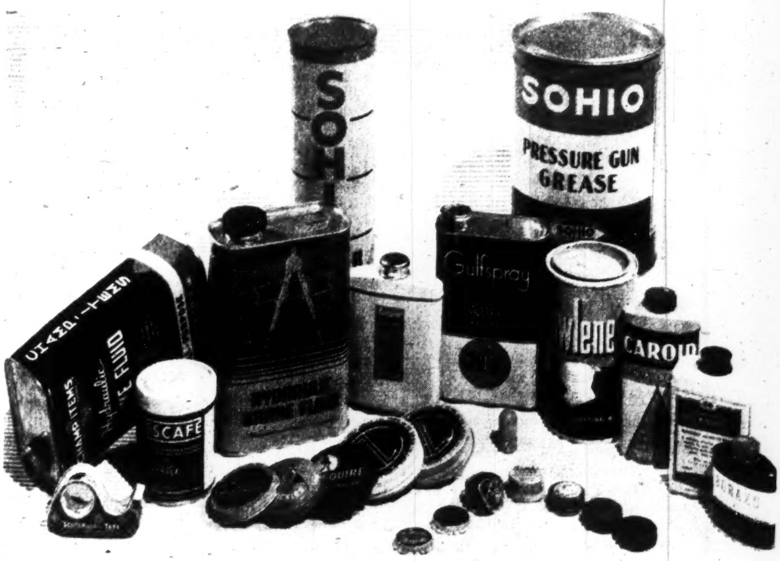

FIG. 20.—Canning Industry: Metal Coated Inside and out With Phenolic, Alkyd or Vinyl Resins, and Formed after Finishing.

Courtesy of Finishes Division of Interchemical Corp.

Sometimes it is just as important to keep the containers from being attacked by the contents as it is to keep the contents from being contaminated by the container. Linings are available that will resist petroleum solvents and greases, sulfonated oils, esters, aromatic hydrocarbons, alcohols, lacquer thinners, plasticizers, terpene products, alkaline soaps and powders, water softeners, acetic anhydride, formaldehyde, glycerin, ferric chloride, and many other corrosive chemicals. The linings make it possible to use low-priced steel containers where otherwise glass, with its attendant boxing and crating, would be required.

These protective sanitary linings are based on synthetic resins, usually of the phenolic or vinyl type. In food cans, the tinplate is given a coating in the flat sheet with a roller coating machine, and the film is baked for 20 to 25 min. at temperatures around 375 to 425 F. The coatings may be colored or transparent.

As with other applications for indusrial finishes, there is a specific sanitary lining that does the best kind of job for any given combination of conditions,

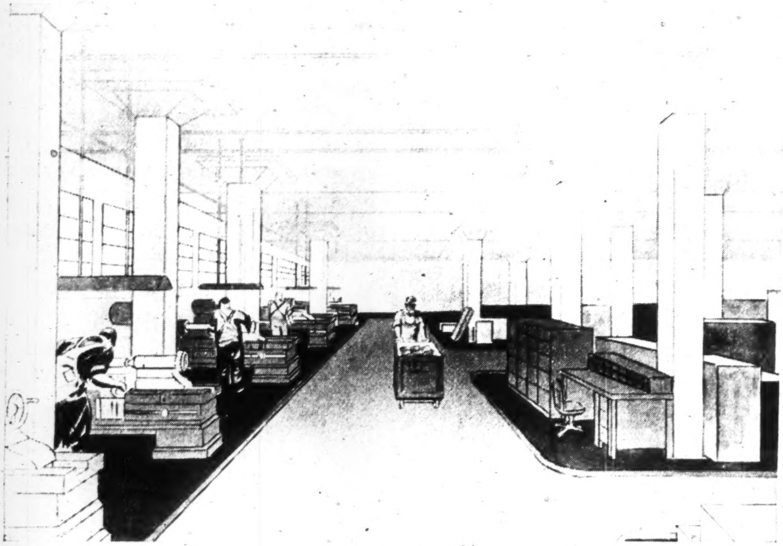

FIG. 21.—Proper Painting Method for Industrial Plant.

Illustrating:

(*a*) Elimination of glare from ceiling, walls and floor.
(*b*) Sectioning off the floor areas.
(*c*) Separation of the critical from the noncritical parts of operating machines.

Courtesy of Pittsburgh Plate Glass Co.

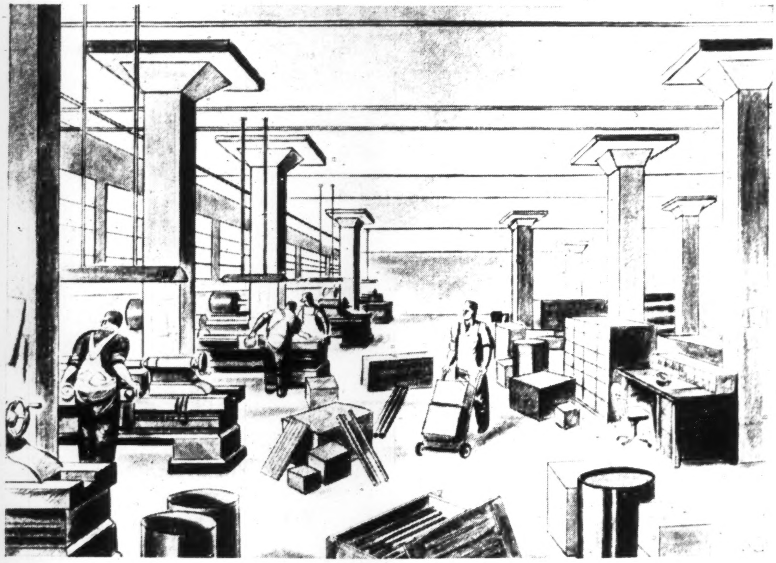

FIG. 22.—Lack of an Organized Painting Scheme.

Courtesy of Pittsburgh Plate Glass Co.

and the experienced metal decorator knows which of these coatings to select to achieve the protection he desires. Figure 20 shows the various types of containers used in the canning industry. It is interesting to note the different degree of forming the coated sheets have to stand to produce the different types of containers.

Thanks to the paint industry, and in particular to the metal decorating technicians, you may consume your canned fruits and vegetables with the utmost confidence, knowing that because of a sanitary can lining they are as pure and wholesome as they were when they were put into the can.

COLOR IN INDUSTRY

The use of colored coatings in industrial plants and on machines involving the use of color to highlight, reveal, and emphasize is discussed at length by Williams (**54**) and by Keshan (**26**). They disclose the gaining popularity of colored paints in all engineering industries.

A good painting program has sound functional value. It has been shown that the proper use of color can reduce eyestrain and fatigue, assist in maintenance by encouraging cleanliness, identify piping and other vital units, improve morale and safety, and actually increase production and accuracy.

The full effects of color values have been achieved by those plants which apply color to their entire environment —walls, ceilings, floors, dollies, jigs, bins, machines, and all other equipment in the vicinity of the operator. The fundamental purpose of coloring is to provide cheerfulness, restfulness, and good visibility. Extensive experiments have shown that proper working conditions are obtained with ceilings that reflect at least 75 per cent of the light rays and walls reflecting 50 to 60 per cent. Although white reflects most light, it is not necessarily wise to coat everything white. Research has shown that white walls and ceilings actually tend to reduce visibility. This loss in visibility is caused by the flood of light from these surfaces which causes the pupils of the eye to contract when viewing the darker machines and work. Eye fatigue can be lessened by having eye-rest surfaces, such as the surrounding walls, coated with the same general tone of color as the worker sees when bending over his work.

In its application to machinery, the first step in the use of color is the separation of the critical from the noncritical parts of the machine and the highlighting of the critical. The critical parts, which include all moving parts and danger spots, are painted with colors designated "focal." Focal colors are designed to focus the worker's attention precisely where it should be. Buff, cream, and light green are widely used for working parts, while orange is selected for the danger spots like electric switches and buttons. The second step is the relegation of noncritical parts to the background. For this purpose a medium shade of green is recommended for machine bodies. Green has a soothing effect on the eye and presents a receding effect which makes working areas painted in buff or cream stand out. Cutting oil-resistant, washable, gloss or semi-gloss paint is recommended for this work. Surfaces painted with this type of coating can be wiped off easily with waste or cleaned with solvent cleaners. Figure 21 illustrates the value of the application of the color scheme discussed in the painting of the machines, walls, floors, and ceilings of an industrial plant, whereas Fig. 22 shows the lack of any color plan. If color plates were used here, the full effect of the color values could be illustrated more effectively. Figure 23 shows the painted sterile areas for Penicillin packing.

A system of piping identification which aids in preventing time wasting and sometimes disastrous confusion in plants has been recommended by the American Standards Association, the National Safety Council, and The American Society of Mechanical Engineers. In this practical method it is advised that piping be painted to match the surrounding areas and that only couplings, unions, valves, or bands be used in identifying colors. This saves in maintenance cost and eliminates gaudy effect of a large number of brightly colored pipes. The color symbols are:

Red—fire-protection equipment
Yellow—gas or dangerous materials (acids)
Blue—steam or water exhaust and protective materials (other than for fire-protection)
Green—airlines and safe products (drinking water)
Purple—extra valuable materials

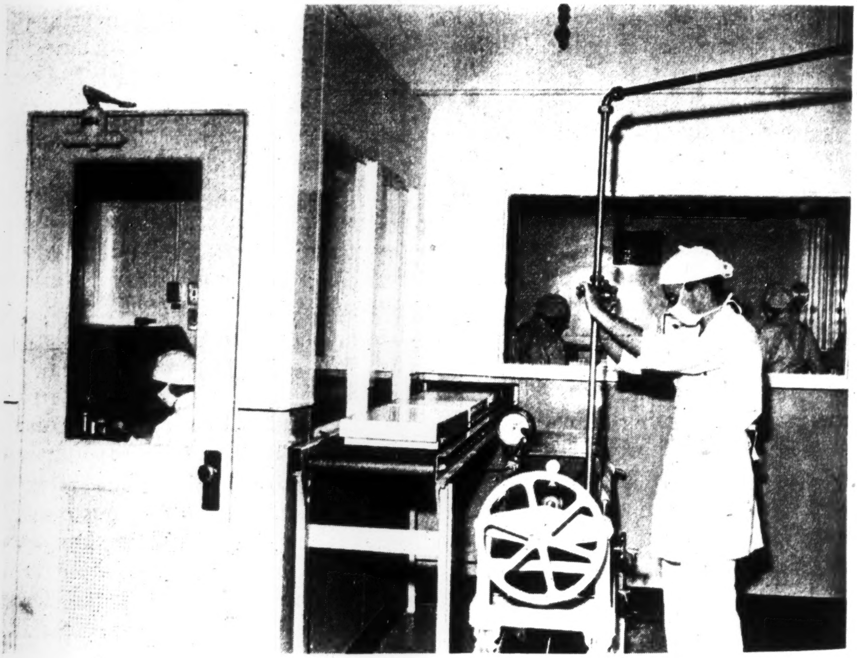

FIG. 23.—Painted Sterile Areas for Penicillin Packing.

Courtesy of Chas. Pfizer & Co., Inc.

As a safety measure, all aisles and traffic lanes are plainly marked. The entire aisle is painted in a color contrasting to the body of the floor, or boundary lines are made of thick stripes of a vivid color. Mobile equipment, such as trucks, conveyors, and cranes, are painted with high visibility colors so that workers cannot fail to notice their approach. Safety color codes are being used widely to prevent accidents and to highlight equipment. A typical one is given herewith:

Safety Color Code Chart

Yellow—(high visibility) to mark trucking equipment, conveyors and aisles at hazard points, platform edges and pits, railings, protruding parts, curbings, floor level changes, chain hoist blocks.
Orange—(alert) to mark parts of machines or equipment that might cut, crush, electrocute: interior surfaces of fuse and power

boxes, machinery guards, pulleys, gears or cutting devices, electric switches.

Green—(safety) to identify and locate first-aid equipment, dispensaries, medicine cabinets, respirators, stretchers, and surgical wagons.

Red—(fire protection) to locate and indicate instruments for combating fire danger: fire hose, extinguishers, alarm stations, and hydrants.

Blue—(precaution) to warn against use of equipment under repair or machinery that should not be started: controls, valves, and compressors.

White—(black or gray) to encourage good housekeeping and orderliness: applied on traffic controls, aisle markings, storage areas, waste bins.

With such a wide application as has been outlined, the color reformation of the factory is complete.

It should also be realized that as this subject is developed further other color schemes may be considered either equally as useful, or more so, than that given in this section.

INFRARED REFLECTING PAINTS

Infrared rays were first observed by Sir William Herschel in 1880. He illustrated his discovery in an experiment, where he placed thermometers beyond the red end of a spectrum produced by the sun's rays passing through a prism, and a rise in temperature indicated the presence of heat rays that were invisible to the human eye.

The extensive fundamental data developed by Ayres and Vesce (**51, 52**) on the spectral characteristics of pigments in the infrared region of the spectrum and formulation of paints with specific infrared reflectance were invaluable in the manufacture of camouflage paints during the war. These infrared reflectance paints towards the latter part of the war began to play a part in the cooling effect produced by the use of them on warships. Some of the Navy's work should be of interest, as its results may also play a part in civilian paints.

The Navy had developed deck paint which had the same reflectivity as the standard Navy paint in the visual part of the spectrum but approached the reflectivity of a white paint in the infrared (heat range) part of the spectrum, for the purpose of determining if the berthing spaces under the weather deck are more comfortable when ships are exposed to solar radiation.

The amount of solar radiation absorbed by a steel weather deck is dependent upon the color of the deck, the wind and spray, the temperature of the air and surrounding objects, the heat capacity of the deck, and its thermal resistance. The energy of solar radiation is approximately 40 per cent in the visual, 5 per cent in the ultraviolet, and 55 per cent in the infrared (heat range) portion of the spectrum. The solar radiation absorbed by most objects with which we are familiar is not accumulated to the extent shown by heavy steel structures, such as a ship. The thin sheet steel of an automobile does not have enough heat capacity to prevent vagrant winds from appreciably reducing temperatures. On board ship there are large masses of steel present such as gun mounts which re-radiate heat to the decks. It is believed that a warship painted in a dark color is more susceptible to infrared radiation than most objects. There is little likelihood of the data given herein being applicable to other objects of less mass.

In port in the tropics there is seldom high wind or spray to dissipate the heat, and the dark-colored deck absorbs the full effect of solar radiation. The deck absorbs a large part of both the 40 per cent of the radiation in the visual spectrum and the 55 per cent in the infrared portion of the spectrum. It is not possible to reduce the amount of solar radiation absorbed from the visible portion of the spectrum since to do this would require altering the color of the decks; therefore, any possible improvement must come from reflecting the 55 per cent of the energy which is in the infrared portion.

The heating effect of solar radiation was amply shown by many tests on the standard ship which showed the average temperature of the weather deck to be 161 F. At the same time the ambient air temperature was only 96 F. After 10:00 a.m. it was not possible for a person to hold a hand to the hot deck for more than a few seconds at a time.

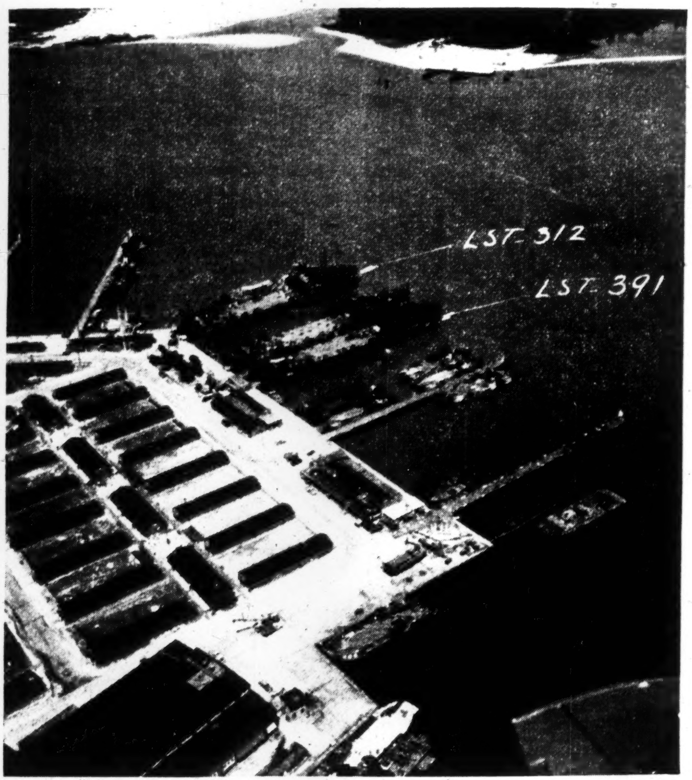

FIG. 24.—Black and White Photos (Visual Perception Photographic) (Panchromatic film).

Courtesy of U. S. Navy

Panel Test at Pennsylvania State College:

The following test was conducted at the Engineering Experiment Station of Pennsylvania State College.

Panels simulating destroyer decking were constructed of 3/16-in. plate on the weather deck of four compartments 67 in. square. The panels were insulated between stiffeners with 3/4-in. fibrous glass insulation board. After confirming that all four compartments were the same thermally, the surface of the panels exposed to the weather was painted with one coat of zinc chromate primer and two coats of deck paint 20B. Care was exercised to finish all compartments the same way throughout and a Pfund paint film thickness gage was used to insure applying approximately equal thicknesses of paint to each panel.

Fifteen thermocouples were attached to each compartment for the determination of various surface and air temperatures. The test data indicated that at

12:30 p.m. the panels coated with infrared reflecting paints were approximately 20 F. cooler than the panel coated with standard deck paint (20B). Since all the test compartments were identical in construction, it was possible to compute the relative heat transfer through the decks. An interpretation of the data developed indicates that under the conditions of this test the heat transmitted through the panel and insulation into the air of the compartment was reduced 33 per cent when the infrared reflecting paint was used as compared to the standard deck paint.

Ship Service Test at Norfolk Navy Yard:

A test was made to determine specifically if painting an LST boat with infrared reflecting paint would reduce the deck temperature and actually make the berthing spaces under the weather deck more comfortable when the ship was exposed to solar radiation. The test was conducted in two parts using two identical LST's: the preliminary part to establish whether or not two LST's were equal thermally when painted alike and exposed to solar radiation; the final part to determine the thermal difference between one ship painted with infrared paint and the other with standard gray paint.

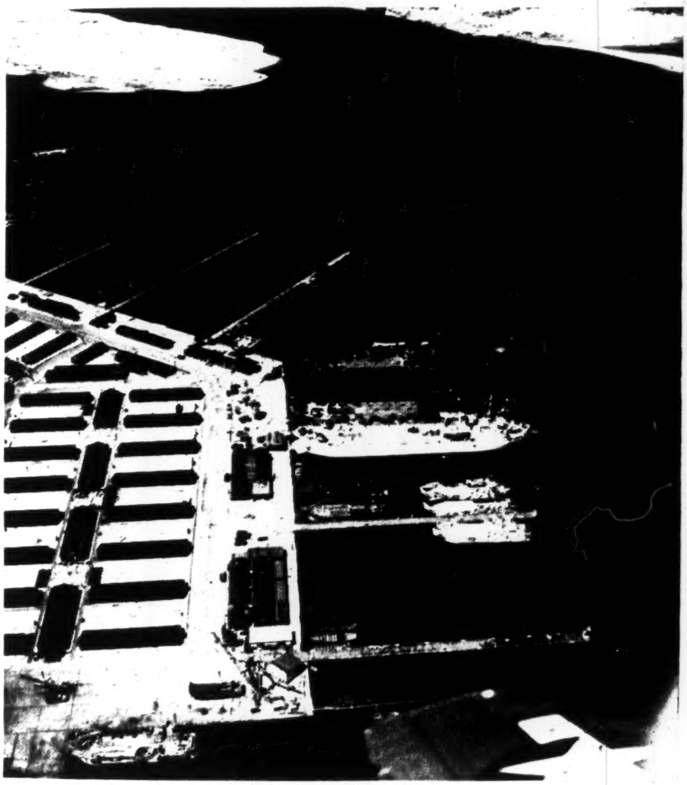

FIG. 25.—Infrared Photograph.

Courtesy of U. S. Navy

For the preliminary part of the test the LST 312 and 391 were painted alike with standard gray paint on March 20 and April 5, 1945, in accordance with camouflage measure 21. The ships were then moved to the Amphibious Training Base at Little Creek, Va., where temperature readings were taken from 9:30 a.m. on April 6 until 4:30 p.m. on April 7, 1945.

For the final part of the test the LST 312 and 391 were then repainted at Norfolk Navy Yard on June 16 and 19, 1945, with one coat of zinc chromate primer No. 84 and one coat of camouflage paint, in accordance with camouflage measure 21, over the entire exterior surfaces of the ship. For the portion of the ships above the water line, standard paints were used on LST 312 and infrared paints were used on LST 391. The two ships were then moved to the Amphibious Training Base at Little Creek, Va., for the final part of the test and moored in the same positions where they were located during the preliminary part of the test. Pictures were taken of the two ships in black and white and infrared as shown in Figs. 24 and 25.

Temperature readings were taken by means of thermocouples at about seven different positions of the LST boats, and in the final test LST 391 (infrared paint) was cooler than LST 312 (standard paint) in every position tested. The average difference in weather deck temperatures at 3:00 p.m. was 13 F. for both days of the final test. The average difference in the air temperature of the compartments tested under the weather deck at 3:00 p.m. was 9 F. for both days of the final test.

One good civilian use of infrared or high solar reflectivity paints was indicated by Lutz (**31**) with regards to decreased evaporation losses in the petroleum industry. He points out that white paints are outstanding in this respect. However, as an approach to optimum condition, he recommends that the body of a storage tank should be painted white with the sides either aluminum or gray.

Of course it should be recognized also that in war activity and even certain civilian use it is not always possible to use white, aluminum, or even gray paints. In those cases colored paints with the highest possible infrared reflectance for the specific color should be used.

Gardner (**22**) has also conducted tests to determine the rise in temperature of benzine contained in small tanks painted in various colors and subjected to the rays of a carbon arc for periods of 15 min. These results show that the rise in temperature was highest for black paint, 34.2 F., whereas for white paint it was 2.7 F., and for aluminum paint 0.7 F. These data are useful only in indicating trends. For practical uses, it should be stressed that solar reflectivity data should be developed under actual solar conditions.

LUMINESCENT COATINGS

Luminescence is the term generally applied to the emission of light not due to heat as contrasted to *incandescence*, which is the term applied to the emission of light due to intense heat. Luminescent light, therefore, is "cold" light.

Luminescent pigments are materials that emit light following the absorption of radiant energy, and the transmutation of such energy to a longer wavelength. Each quantum of photons raises an electron to an upper energy level from which the electron returns to its equilibrium state, either immediately or over a period of time, with the simultaneous release of the absorbed energy in the form of visible, colored light. The emitted light is always of longer wavelength than the radiant energy absorbed in the molecule.

If the electrons return immediately to their equilibrium state, the luminescent pigment is called "fluorescent." A fluorescent pigment, therefore, emits light only during the time of exposure to an exciting light source.

If, however, at least a portion of the electrons return to the state of equilibrium only after a period of time has elapsed, the luminescent pigment is said to be "phosphorescent." Therefore, a phosphorescent pigment not only emits light during exposure to an exciting light, but continues to glow in the dark after the exciting light is extinguished. The afterglow ranges from 30 min. to 10 to 12 hr. and more, depending upon the pigment used.

Exciting Light Sources:

Fluorescent pigments are excited by light sources that radiate very little, or no, visible energy if their fluorescence is to be fully appreciated. Many such lights, equipped with filter to exclude visible energy, are available—argon glow lamps, fluorescent lamps, and high-pressure mercury arc lamps, available in a wide range of wattage sizes.

Phosphorescent pigments, when used only for the afterglow property, may be excited to phosphorescence by sunlight or daylight, artificial visible light, or "black" light.

Processing:

Fluorescent materials may be divided into two groups, depending upon their chemical composition: organic and inorganic.

The organic materials are chiefly synthetic dyes, dye intermediates, and metallic salts of dye intermediates. The dyes include rhodamin, eosin, and flavin, which fluoresce brilliantly when prepared into lacquers, printing inks, and paper coatings. Such dyes, however, have relatively poor fastness to sunlight or daylight.

Somewhat more stable materials may be prepared by precipitating the dyes on suitable inorganic base materials to form lakes.

A number of dye intermediates show fairly strong fluorescence, and, in many cases, the fluorescence can be further increased by preparing metallic salts from them. An outstanding example is the zinc salt of 8-hydroxyquinolin which is considerably more stable on exposure to sunlight than the organic dyes.

The commercially useful inorganic fluorescent pigments are limited in number, and they are generally zinc sulfides or combinations of zinc and cadmium sulfides. Although somewhat larger in particle size than regular paint pigments, they may be ground on roller mills to make excellent paints. These pigments are also suitable for processing into printing inks, silk screen process paints, and paper and textile coatings, and for incorporation in all water-clear types of plastics.

Fluorescent pigments are relatively stable but, under certain conditions of light and moisture, they are subject to a photochemical change called "light darkening." When properly prepared in suitable vehicles, fluorescent paints will give satisfactory service in outdoor applications.

Phosphorescent materials are always inorganic pigments, there being no known organic materials which possess phosphorescent (afterglow) properties.

Phosphorescent pigments of short-afterglow type are also zinc sulfides or zinc-cadmium sulfides which, like the fluorescent pigments, are relatively stable. They are coarser in particle size but are suitable for processing into paints as well as coatings, silk screen process paints, and plastics.

Phosphorescent pigments of the long-afterglow type are calcium and strontium sulfides. These pigments are quite coarse and should not be subjected to milling or grinding but should be stirred into the vehicle. They are best adapted to spray paints, silk screen process paints, paper coatings, and certain plastics.

Since these long-afterglow pigments are sensitive to moisture, care in the selection of the proper vehicle is generally necessary and, for outdoor paint applications, a top coat of clear vehicle is recommended.

While priming coats are not absolutely necessary, for best results—that is, highest luminescent intensity—it is advisable to apply luminescent paint (both fluorescent and phosphorescent paint) over a white priming coat free of heavy base metals.

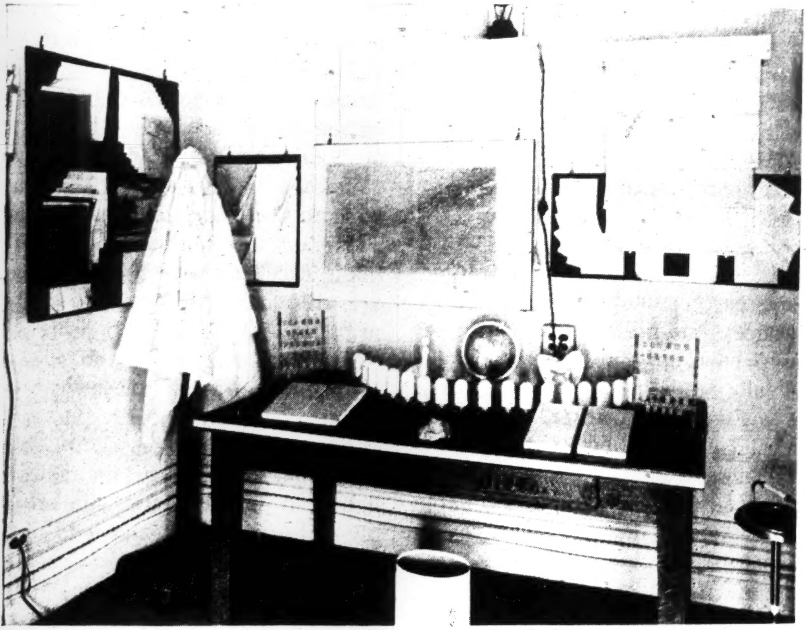

FIG. 26. Display of Fluorescent Materials.

Wall Area (left to right)
- Panel with fluorescent papers (filled papers) printed with halftone of U. S. Capitol Building, Washington, D. C., and maps and charts. (Dark-toned papers are regular papers printed with fluorescent lithographic inks.)
- Two swatches of printed satin (on stand), with (left) yellow fluorescent, and (right) green phosphorescent pigments.
- Panel of baked-coated papers, (left half) different colors of fluorescent and (right) phosphorescent pigments in several colors.
- Navigational map, printed with regular map lithographic inks on (left half) pre-war paper (non-fluorescent), and (right half) the same type of map paper filled with fluorescent pigment (yellow).
- Shades of coated papers (top to bottom) blue phosphorescent, green phosphorescent, red fluorescent, and yellow fluorescent. The blue phosphorescent and red fluorescent colors were affected by the yellow filter used on camera lens in taking these pictures.
- Under paper shades, a panel of safety papers, printed with fluorescent inks, and (center of panel) with regular inks on filled fluorescent paper.

Table Area (left to right)
- Two painted panels (blue and yellow fluorescent).
- Behind panels, (on rack) 3 radio dials, silk-screened with fluorescent paints.
- Glass jars of fluorescent and phosphorescent pigments.
- Behind jars—Carstairs (whiskey) seal, World Atlas, and Eagle Pencil Company eagle—all phosphorescent painted.
- Three more fluorescent silk-screen radio dials.
- Front of dials, painted panels in green and blue phosphorescent paints.
- Center of table—Piece of Willemite (zinc) ore, fluoresces green.
- Phosphorescent tape edging table.
- Phosphorescent coated paper basket under table.

Courtesy of New Jersey Zinc Co.

Colors Available:

While fluorescent pigments are available in colors covering the spectrum from blue to red, the phosphorescent colors are limited to greens and an orange-yellow for the short-afterglow phosphorescent pigments, and to several blues and blue-greens for the long-afterglow types. The daylight colors generally are different from the luminescent colors, but some modification may be made with the use of very small amounts of suitable dyes or color pigments—and this is being done commercially. Too much coloring material should not be used, however, as such materials tend to screen out or mask the luminescent light.

Applications:

Fluorescent Paints.—Interior decorative effects, displays and advertising, radio dials and decoration, and automotive and aviation instrument dials and accessories probably cover the major fields where fluorescent paints will find most interesting applications. Glowing color will be introduced into theaters, night clubs, cocktail lounges, and wherever else darkness or semi-darkness prevails. Under such conditions, fluorescent displays under "black" light also are colorful, unusual, and interesting. (See Figs. 26 and 27.)

A very interesting application for fluorescent pigments and "black" light is offered by television, the reception of which is most effective if viewed in darkness or at least semi-darkness. Here, radio dials and decorations, painted wall decorations, probably with fluorescent drapes and upholstery, will bring colorful decoration into an otherwise cold, dark, unattractive interior. And, of course, safety of movement within the room will be increased.

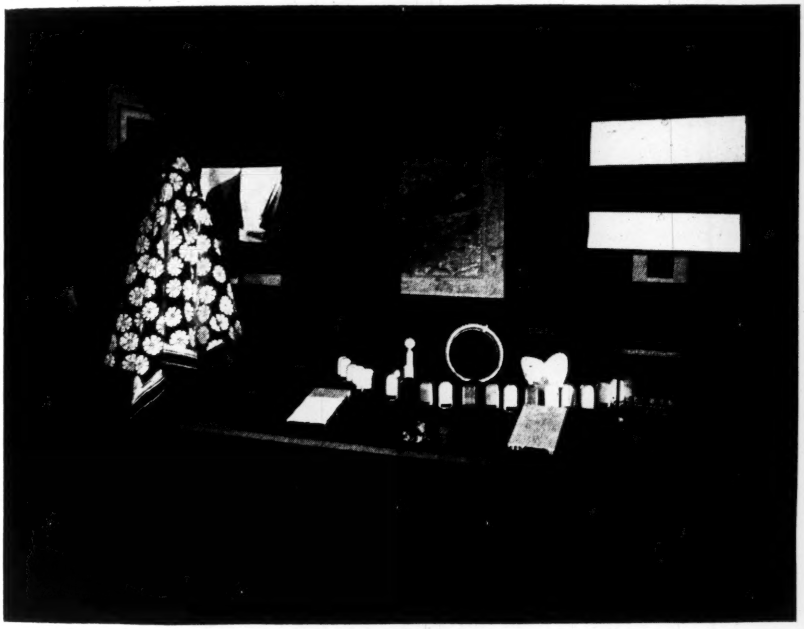

FIG. 27.—Black Light Version of Fig. 26.

Courtesy of New Jersey Zinc Co.

Fluorescent aviation and automotive instrument dials provide ideal illumination for more comfortable night flying

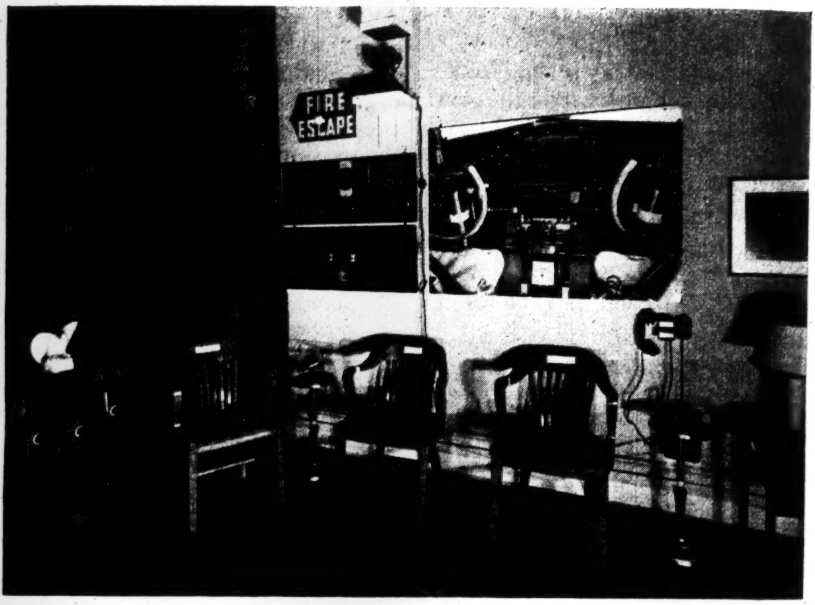

FIG. 28.—Display of Fluorescent Materials. *Courtesy of New Jersey Zinc Co.*

Wall Area (left to right)

(Top)—"Fire Escape" sign—phosphorescent.
(Below Sign)—Panel of fluorescent navigational instruments and correction charts used on all war planes.
(2nd Below)—Panel of instrument dials on war planes, utilizing both fluorescence and phosphorescence.
(Center)—Photo of instrument panel in Flying Fortress. Dials are fluorescent, while instruction plates and group markings around dials are phosphorescent.
(Extreme right)—Picture frame contains phosphorescent decalcomania instruction panel for planes.

Floor Area (left to right)

Radio with fluorescent dials (black light 6-watt 360 BL inside of set) and phosphorescent rings around control knobs.
Phosphorescent eagle on radio.
Chairs, ash trays, and telephone marked with phosphorescent tape.
Fluorescent rug on floor.
Phosphorescent plastic lamp shade and pedestal.
Phosphorescent tape on baseboard behind chairs.

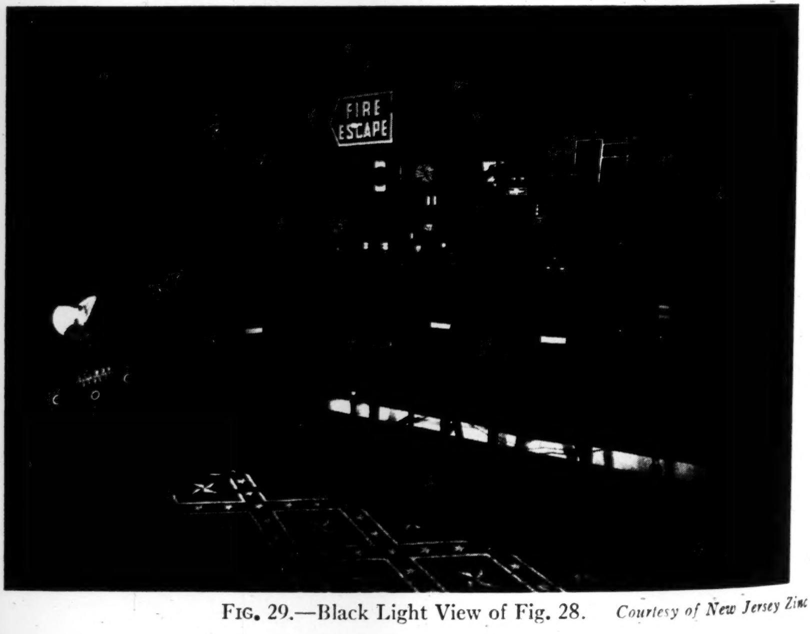

FIG. 29.—Black Light View of Fig. 28. *Courtesy of New Jersey Zinc Co.*

and driving, and for greater safety. The fluorescent paint or "black" light system was used on combat planes during the war, and it is interesting to know that all of TWA's Stratoliners and DC-4 (Douglas Transport) planes are similarly equipped for peacetime travel.

Fluorescent paints, inks, and papers also can be used as anti-counterfeit measures—for making safety papers or as marking devices.

Phosphorescent Paints.—Phosphorescent paint probably will find its greatest use in safety applications, for example, as emergency light sources, including exit and instructional signs, markers for controls, communications, alarm systems, and fire-fighting equipment. Other items that are more easily located in the dark when marked with phosphorescent paints include electric switch plates, switch boxes, and outlet plates; clock dials; cord pulls; flashlights; house numbers; stair treads and railings; safety aid kits, etc. (See Figs. 28 and 29.) If a satisfactory moisture-resistant, tough, flexible, abrasion-resistant vehicle is developed, traffic line paints may be developed.

Undoubtedly, luminescent plastics, plastic adhesive tapes, and coated papers will offer stiff competition to luminescent paints in many, if not all, applications for these colorful, glowing materials.

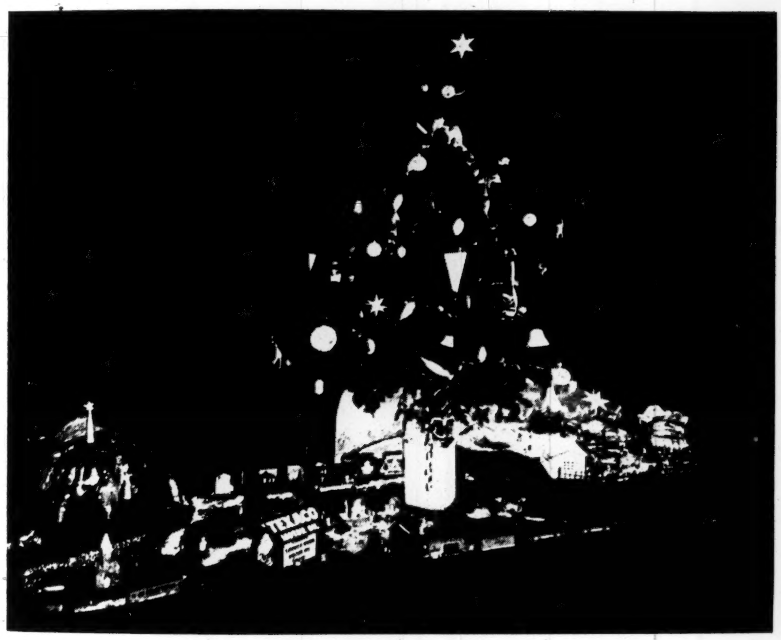

FIG. 30.—Luminescent Christmas Tree.

Courtesy of New Jersey Zinc Co.

Luminescent materials can also brighten our Christmas, as illustrated by George Stutz's Christmas Tree (see Fig. 30).

MARINE PAINTS

Introduction:

Marine environments, particularly in salt water, present exposure conditions which lead to severe corrosion of steel.

Marine fouling is an age-old problem. It has been estimated (1) that fouling on ship bottoms costs the shipping industry of the United States alone at least $100,000,000 per year. Adamson (1), Burns, *et al.* (10) have indicated that fouling can result in loss of the speed of a ship to approximately 50 per cent and an increase in fuel consumption of at least 40 per cent. In extreme cases large vessels have collected several hundred tons of fouling having an average thickness of over 6 in. Burns (1) has indicated that it has been the practice of the British Admiralty, for design purpose, to allow for an increase of frictional resistance of $\frac{1}{4}$ per cent per day out of dock in temperate waters and $\frac{1}{2}$ per cent per day out of dock in tropical waters.

It is no wonder, then, that the painting of ship bottoms alone has attracted the serious attention of paint chemists, chemical engineers, and others to produce protective coatings which would increase the engineering efficiency of all kinds of vessels. In this short section a general discussion of painting steel ships will be presented, whereas, because of special interest, infrared reflectance and hot plastic paints are presented separately.

Ship Areas:

A ship is divided roughly into three areas: the bottom or hull portion of the ship, which in service is continually immersed in the salt water; the boot-topping area, which is the hull portion from the light load to the heavy load line or that portion of the hull which is alternately immersed in the sea and air; and the topside area, which is that above the heavy load line including the superstructure. The interior metal surfaces of ships represent a somewhat less severe condition than the exterior topside but are generally treated similarly to the latter.

Ship Painting:

The topside or superstructure area of a ship must be painted not only for protection but also for appearance. The corrosive influence of sun and salt spray combine to give an unusually severe environment. Only the best primers and topcoats applied to a clean surface will give adequate performance.

The boot-topping paints must be formulated to provide sufficient flexibility to withstand the alternate wetting and drying to which these coatings are subjected. The primer for this type of surface often varies from that used on the topside but the main difference will be found in the topcoat. The vehicle should provide a film with good impermeability and one which will retain flexibility under these conditions. The boot-topping paints must also withstand the various surface conditions encountered in harbors and sea lanes such as oil slick, harbor gases, refuse, driftwood, ice, algae, and slime.

The ship bottom paints for use on the completely submerged portion of the ship (below the light load line) present a third problem. Not only must protection against corrosion be provided but also, as already indicated, protection against the detrimental effect of marine fouling organisms. The fouling organisms differ somewhat in nature and extent, depending upon the climate in which they are found. However, they can be classified as slimes, which are minute diatoms and other unicellular organisms; seaweed or grasses; algae; true animal fouling, which can be divided into those which produce hard shells (barnacles, tube worms, and hydroids) and those which do not (sponges, sea squirts, and certain polyzoans). The hard-shell animals are the most troublesome since even when dead they leave their shell firmly attached to the ship and also because they bore through the paint film and expose the metal to corrosion. Fresh water has a cleaning action on fouled ships since it kills the sea life. Ports which contain a fresh water inlet are known as scouring ports since a ship which lies to in one of these ports will be partially freed of fouling. The only practical way to minimize or prevent fouling is to use paints containing copper and mercury salts which, as they leach out, poison the organisms that attempt to attach themselves to the ships.

The use of mercury and copper salts in the antifouling coats calls for an additional problem in the undercoats. Whereas the antifouling topcoats provide protection to the corrosion-preventing undercoats, they can in themselves cause corrosion if they come in direct contact with the steel. This is due to the fact that metallic copper can be deposited which would provide the basis for a galvanic couple leading to severe electrochemical corrosion. The anticorrosive primer must, therefore, possess a high degree of impermeability.

To attain this high degree of impermeability an increase in film thickness is necessary, achieved by the application of additional coats of anticorrosive paint.

Wooden ships require the same attention as steel vessels with respect to the application of properly designed protective coatings for best engineering efficiency.

Locks and Dams:

The problem of protecting metal work exposed to fresh or river water on the great network of locks and dams interspersed throughout the Midwest and Pacific Coast is also difficult. Since maintenance operations are costly, especially when gates must be inactivated in order to repair or paint, much care in initial application of protective coatings is mandatory. The U. S. Corps of Engineers has given the subject considerable study and thought, with the result that methods of surface preparation and painting have been evolved which insure a maximum degree of protection for metal work exposed to varying conditions.

The destructive forces of fresh water are not confined to the action of the water but also include the problem of erosion of the paint and metal by the salt and sand which the rivers carry. This combination is much more destructive than either one alone. There is also the additional matter of acid pollution in many of the fresh water streams against which river boats, locks, dams, etc., must be shielded. The sources of this acid pollution are water waste from coal, steel, and other mines; waste pickle solution; and discharge from iron and steel rolling mills, paper mills, tanneries, oil refineries, etc. Paints must therefore be designed to combat this erosion by being harder and more impermeable than is necessary for atmospheric paints.

In the painting of locks and dams, as with all other surfaces, proper preparation of surface is considered equal in importance to the protective coatings system chosen. Whenever possible and practical, sand or shot blasting of the steel surface is recommended in order to remove all insecure or corrosion-activating bases to which a protective coating would be applied. Such preparation cleans the object down to bright metal. It is then advisable that, without delay, the bright steel be given a chemical rust-inhibiting wash of the phosphate-forming types. Such a procedure is required for two reasons: first, because the deposited coating serves as a temporary protective barrier between the highly reactive metal and the surrounding atmosphere until paint can be applied, and second, because it has been found that the life of a paint system may be materially increased when such a base is present. Blistering and rust creepage are minimized and adhesion of the priming coat is improved.

The paint system may be varied depending upon the severity of the intended exposure and the type of paints chosen. Probably the best system specified for this type of exposure includes the use of two coats of the primer paint followed by two coats of a finish coat paint, that is, a four-coat system.

TROPICALIZATION OF PAINTS

The problem of tropicalization of equipment became acute as the theater of war shifted to the South Pacific areas. It will remain of importance to the United States as long as we hold bases on the islands throughout the Central and Southern Pacific area where high temperature and high relative humidity prevail. The Philippines have always been faced with a similar problem. The problem is, therefore, an old one, but the need for its solution never before was so great as when tremendous quantities of supplies and equipment began to pour into the Pacific bases. Communications and electronic equipment were especially hard hit. The walkie-talkie of the Signal Corps had a life of only a few hours as fungus would grow upon the very sensitive coils and short circuit the sets by holding moisture in the mycelium of the fungus.

Among the workers in this field there is considerable discussion of the necessity of the presence of a fungicide in an insulating varnish. Some say that if a tung oil-phenolic varnish of known high-moisture resistance is used, a fungicide is unnecessary and would even deteriorate the varnish. Studies are being made at present on the resistance of the coating to all types of fungi without fungicides present and also on the comparative durability of the varnishes with and without fungicides.

In many instances impregnating compounds and paints for fabrics have been designed to resist the action of *Chaetonium Globosum*, an organism which destroys cellulose. Very little or no protection at all has been used against *Aspergillus* and surface-type fungi because it is said that they can be brushed off with no damage to the material. This is possibly true in certain instances, but in a number of cases it has been found that the surface-growing fungi actually destroy the fungicide used to prevent the cellulose-destroying fungi, and after *Aspergillus* has disappeared *Chaetonium* will follow and destroy the fabric. This type of failure points out the fallacy of testing against specific organisms. The only tests found to give comparative results with actual exposure have been those in a "tropical" chamber with controlled climatic conditions and the presence of many tropical species of fungi. This was clearly illustrated by the recent exposure of a commercial white paint which the manufacturer claimed was fungus-proof. The paint had been proofed against certain definite types of fungi, but when coated out upon wall board, wood, etc., and exposed in the tropical chamber, showed within 48 hr. a luxuriant growth of numerous species of fungi.

Exposures of paints containing various fungicides have been made in several different ways. In some instances the paints are coated out on panels, usually wood, and dried under ideal conditions of humidity and temperature in an atmosphere free of fungi spores and then shipped to the southern part of this country or the Canal Zone, which regions abound in mildew growth. This type of exposure is unsatisfactory for two apparent reasons: the paint is allowed to dry and thereby become resistant to spore infection before exposure, and, the atmospheric conditions of the tropics do not have a chance to exert an influence on the paint film during the drying stages.

Another type of exposure which is a little more satisfactory than the previous one is the actual coating of panels in the tropical climates. If the coating of panels with paint containing fungicides can be accomplished during a season when the fungi spores are abundant, the humidity high, and the climatic conditions ideal for mildew growth, the tests will be held at ideal conditions. However, if the weather is warm, somewhat dry, and adverse to the sporation of mildew, the application of the paint to the panels might as well have been carried out in the laboratory under almost ideal conditions. Tests for determining the efficiency of fungicides in paint should be carried out in a tropical chamber where conditions are ideal for the growth of mildew at all times and where a high temperature and high humidity prevail. The fungicides should preferably be incorporated into a paint by first dissolving the fungicide in a volatile solvent. Single pigment paints such as white lead or titanium oxide are better for such tests. The paints, panels, and brushes should be seasoned in the tropical chamber for a minimum of 24 hr. Each paint should be applied with a new brush to prevent contamination. This will present most rigid conditions under which the paint may be exposed to evaluate the fungicides. Such tests are being conducted by the Engineer Board, with the fungicides developed during the war. These tests will be fundamental and will give results that can be used in additional tests where paints of mixed pigment and various types of oils will be used.

Some of the fungicides which have been successfully incorporated in paints are mercurial and organic mercurial salts, chlorinated and nitrated phenols, copper, organic and inorganic salts, and certain organic materials such as salicylanilide.

MISCELLANEOUS PLANT USES

For a titanium pigment plant, Robertson (**43**) has pointed out the following important uses:

1. In the operation of some electrolytic cells, where chlorine is produced in the presence of ferrous chloride and it is necessary to support the cathodes in wooden frames, the average operating life of these frames without protection is about 3 months. When coated with a vinyl chloride solution paint, however, the life of the frame is more than doubled, making the difference between an economically impractical and an economically practical operation.
2. In another instance it is necessary to use a wooden basket (yellow pine) submerged for 15-hr. periods in a solution containing ferrous sulfate and about 17 per cent sulfuric acid coupled with a titanium radical at an average temperature of about 150 F. Without protection these frames have a life of only a few weeks, which is increased to 5 months when they are coated with dimethylol urea and urea.
3. In the protection of the exterior of a number of large steel tanks from atmospheric corrosion where sulfuric acid mists are present in the atmosphere, an anticorrosive primer, followed by asphalt-base coatings or chlorinated rubber-base paints, has been found satisfactory. The finish coat gives better service than the former.

McMullen (**35**) has indicated an interesting engineering aspect of organic coating in the use of two Pomona pumps for extracting mine water from some 300 ft. below ground. In this particular case the mine was closed for several months. During that time the zinc sulfide oxidized to zinc sulfate, acidifying the mine water to a pH of 1.5. When the mine was open two pumps made of best Tobin bronze lowered to water level corroded in less than 30 hr. Holes had developed in the impellers.

New pumps of supposedly corrosion-resistant bronze also corroded in less than 30 hr. Stainless steel at this time could not be secured. A high-bake phenol-formaldehyde base paint, pigmented with a high percentage of blue lead, was applied to a new set of impellers, and baked at 400 F. Four coats were applied. At the end of a month's operation, the only failure occurred at the extreme sharp edge of the impeller. This was metallized with stainless steel, and operations continued for several months satisfactorily. If the water level is kept low in these mines, the pH does rise to nearly 7, thus minimizing the danger of general corrosion.

If the pump impellers had not been protected by means of paint, the mine could not have been opened. Each of these pumps costs approximately $2500.

HOT PLASTIC PAINT

Definition and History:

At ordinary temperature so-called hot plastic paint is a solid in which pigment is mechanically dispersed. It is applied in a molten condition without the aid of a solvent, and dries to a solid by cooling —not requiring oxidation or polymerization. The pigment volume concentration is generally around 10 per cent. The best-known application of hot plastic paint is in the protection of Navy ship bottoms. The history of hot plastic paint goes back to the nineteenth century when, in 1899, an Italian hot plastic paint for brushing, with trade name "Moravian," was tested at the Norfolk Navy Yard and found to have unusual durability. In 1901 it was brushed on the U. S. S. Wagneta, and the paint required only retouching at the next dry docking 6 months later. The Chemical Warfare Service of the Army at the Edgewood Arsenal developed a hot plastic in 1924 which was manufactured and tested at the Norfolk Navy Yard and found to be unsatisfactory because of inadequate adhesion. In 1932, at the Mare Island Navy Yard, Moravian hot plastic was applied by brush on the U. S. S. Milwaukee, and some of this paint was still in good condition 30 months later. In 1938, the Paint Laboratory at the Mare Island Navy Yard announced the development of the first sprayable hot plastic. At the outbreak of war in the Pacific, it was realized that some ships might be required to remain in extremely fouling water for the duration without an opportunity for repainting, and it would, therefore, be necessary for the bottom paints to protect the hull for 18 months or more. Generally speaking, the water impermeability of practical bottom paints is proportional to the film thickness applied; and, since no practical method, other than hot plastic, was known of applying extremely thick films, it was considered the best possibility. A few service tests indicated that hot plastic bottom paint would last 18 months, which was considerably longer than the durability of orthodox solvent paints. Therefore, in 1943, it was decided to standardize hot plastic for steel bottoms.

Research, development, and production were carried on simultaneously, and by 1945 over a thousand formulations were under test. So-called cold plastic ship bottom paint is the same as an orthodox solvent containing paint, except total solids are higher, generally above 80 to 90 per cent by weight. The resultant viscosity usually runs over 100 Krebs units, and it should be formulated to have a thixotropic body to eliminate sagging and running when applied to a wet thickness of 6 mils. Because of its high viscosity, it is necessary to use over 90 lb. air pressure in the tank for spraying, and it is sometimes necessary to warm the paint during the winter. There is a tendency for painters to thin cold plastic paint to around 75 Krebs units viscosity, with the result that it gives a thin film and is no more effective than orthodox paints. In this discussion, the classification "plastic coatings" will be confined to those which do not require the use of volatile solvents, and since this requirement is not met by the so-called cold plastics, they will not be considered further.

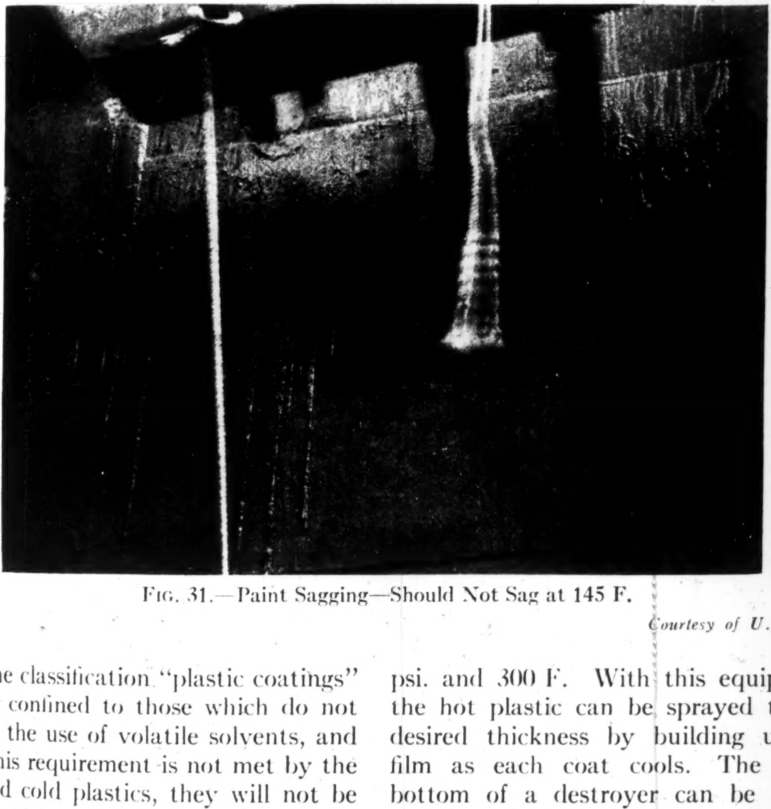

FIG. 31.—Paint Sagging—Should Not Sag at 145 F.

Courtesy of U. S. Navy

Application:

The application of hot plastic is considerably more exacting than that of orthodox paints. Ships for repainting should be prepared by sand-blasting to bright steel, and by spraying with a 2 to 5 per cent phosphoric acid solution, and by priming with an anti-corrosive 3 or more mils thick. The hot plastic should be sprayed to a thickness of approximately 30 mils, using a double pass of the gun, which amounts to over ten times the film thickness of orthodox bottom paints. The solid paint is melted in large heating kettles and transferred to electrically heated pressure kettles, which generally operate at 90 psi. and 300 F. With this equipment, the hot plastic can be sprayed to any desired thickness by building up the film as each coat cools. The entire bottom of a destroyer can be coated with hot plastic by an experienced crew in less than two hours.

General Requirements:

The testing of many different types of hot plastic has led to the following general requirements: The viscosity of the paint should not be less than 1.5 and higher than 2.8 poises at temperature of application—generally between 275 and 300 F.

If copper is the antifouling ingredient, the leaching rate of the matrix and that of the copper pigment should be balanced to permit copper to leach from the film at a rate of, at least, 10 mg. per 1000 sq. cm. per day when the film is immersed in salt water.

The paint should not sag or creep when the surface upon which it is applied is heated to 140 F. Figure 31 shows the sagging which took place on a newly painted hull when a paint of 125 F. sag point was exposed to solar radiation.

The paint should show no plastic flow when tested on a 30-in. diameter spinning cylinder, moving at a peripheral speed of 30 knots in water at 85 F.

This test is to simulate the plastic flow developed on an originally smooth bottom of the ship as it makes a run through tropical waters at over 30 knots.

Degree of Success:

During the early development, stories were told of the hot plastic flying off into the water at the first full power run after painting. Detailed docking reports on the condition of all bottoms are now maintained by the Bureau of Ships, and 18 months is not unusual for the durability of hot plastic, which compares with 9 months for orthodox paints. In general, the anti-fouling properties outlive the corrosion resistance of hot plastic, and when corrosion begins it takes the form of small hair cracks. The degree of success obtained with hot plastic has been proportional to the quality of the surface preparation, which was well demonstrated by a test in which the one side of a ship was sandblasted and the other loosely hand wirebrushed. The ship was then primed with anti-corrosive and quartered, and hot plastic was compared with the ortho-

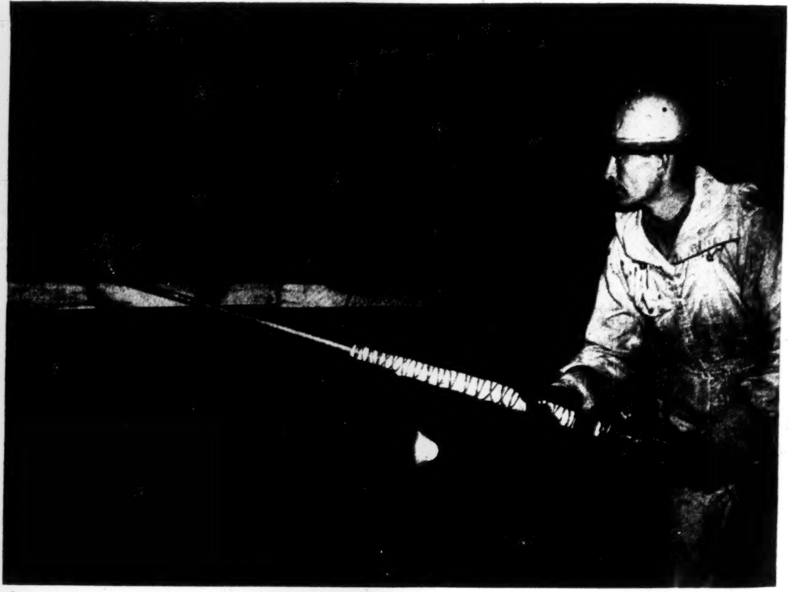

FIG. 32.—Extension Spray Gun.

Courtesy of U. S. Navy

dox type. After 8 months in salt water, the hot plastic, compared with the orthodox type, gave decidedly inferior corrosion resistance over improperly prepared steel and decidedly superior corrosion resistance over the sandblasted surface. The thick impervious layer of hot plastic which prevents moisture from penetrating also inhibits the escape of moisture trapped under the film during painting, and this is the basic reason why it is not durable over a poorly prepared surface. When trapped moisture or the products of corrosion break through a thin orthodox film, nothing more than a pinhole may result, but when this occurs to hot plastic a large area may be torn from the hull.

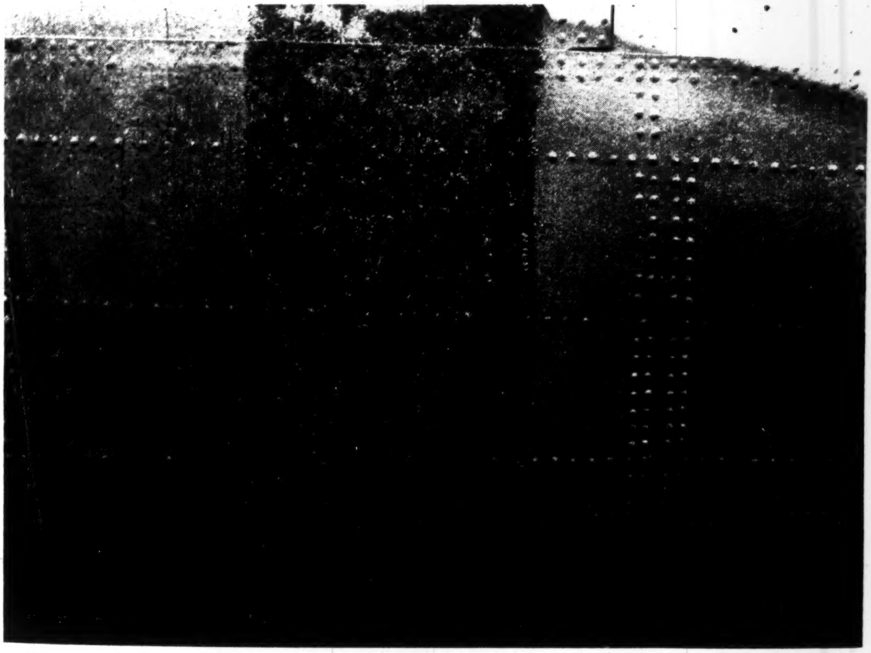

FIG. 33.—Fouling Organisms Growth of Three Paints on Hull of Ship Waterborne Eight Months

Cold Plastic, left—Old 15RC, center—Hot Plastic, right.

Courtesy of U. S. Navy

The success of hot plastic has been dependent upon the development of adequate equipment for surface preparation and application. This development has taken place in the last five years, and in large measure is due to helpful suggestions made by workers using the equipment. At the present time, the development of equipment is lagging far behind the formulation of the paint. An example is the hand gun, which, when used on the flats, produces a fan one end of which generally travels two or three times as far as the other to reach the hull.

The paint traveling in the long end of the fan cools and hits the hull in a partially solidified condition, which will result in a porous film through which water can pass. This difficulty is responsible for the rusty condition of most flats painted with hot plastic. An experimental extension gun which facilitates the elimination of porous spots is shown in Fig. 32.

A 10-gal. single-container pressure tank is standard, and, as a result, the operator is inactive during the refilling operation. However, a double-action pressure kettle which permits the operator to work without interruption is under development. Hot plastic has demonstrated protection against corrosion and fouling on panels for periods of over 4 yr. However, on ships in action good protection has been attained for 18 months, and this only on hulls which have been cleaned to bright metal and primed with two or more coats of anticorrosive. This is approximately 6 to 9 months longer than the orthodox type will last. Figure 33 shows the durability of three different paints, cold plastic, old 15 RC, and hot plastic painted on the hull of a ship and waterborne 8 months. Figure 34 shows the large growth of fouling organisms on the 15 RC paint against little growth on hot plastic paint.

It is difficult to compare the costs of hot plastic and orthodox painting because the two methods require different types of application and lengths of time in dry dock. However, making the assumptions enumerated below, it is possible to make a cost comparison based on keeping the bottom of a liberty ship in good condition over a 3-yr period. This amounts to $18,000 when using hot plastic as compared with $15,000 using the orthodox type. In the $18,000 estimate for hot plastic it is assumed that the surface will be prepared by sandblasting and phosphoric acid treated, and two coats of anticorrosive and one coat of hot plastic will be sprayed. Two days dry docking time will be needed for the initial paint job, and no further painting will be required throughout the 3-yr. period. In the estimate of $15,000 for the orthodox paint system it is assumed that the surface will be prepared by mechanical wirebrushing, one day will be required to complete the job, each 9 months (more often for Pacific waters) during the 3-yr. period the ship will be given two coats of anticorrosive and one of antifouling. Therefore, it can be concluded that hot plastic is not economically competitive with orthodox bottom paint in this particular case.

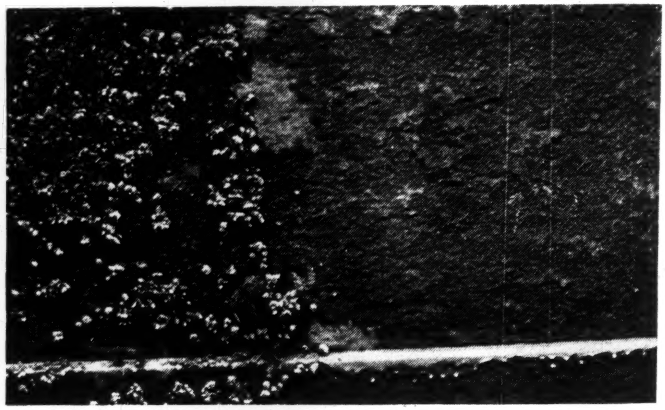

FIG. 34.—Fouling Organisms Growth of Two Paints on Hull of Ship Waterborne 17 Months. Old 15RC, left—Hot Plastic, right.

Courtesy of U. S. Na

Future Research:

Many testing methods new to the

paint industry have been adopted for hot plastic research in order to elevate the work from the "witches' brew" stage to a scientific basis. Some of the methods are leaching rate of toxics and matrices, plastic flow resistance, electrical measurement of the initial time and area of corrosion, determining the type of corrosion, measurements of osmotic and endosmotic water pressure in films, and the use of randomized replicated methods of designing tests.

The hot plastic bottom paint system of the future will probably be considerably different from today's. In place of an anticorrosive which has a film thickness of one-tenth that of the hot plastic, a thicker anticorrosive will probably be used. Five or ten mils of antifouling paint would be adequate for fouling resistance over a 3-yr. period. However, experience has shown that for adequate corrosion resistance, it was necessary to use 30 mils of hot plastic paint. It is not logical to use a copper-containing paint which is conducive to galvanic corrosion for inhibiting corrosion. However, no satisfactory thick anticorrosive is in use. Many hot plastic anticorrosives have been tested, but none has given adequate adhesion when used over the dust and moisture generally present on hulls. A so-called hot solvent anticorrosive paint has been developed which is a compromise between hot plastic and the ordinary solvent type paint, for it is melted at the time of application and contains enough solvent to permit the matrix to penetrate through dust and to orient itself at the steel surface. It has been possible to apply hot solvent anticorrosive paints of adequate adhesion to a thickness of 70 mils without sagging. It is believed that eventually some such compromise thick film anticorrosive will be used followed by a single coat of antifouling paint.

The equipment developed at great cost to the Navy offers a challenge to the paint industry. Large amounts of the equipment have been distributed throughout the ship yards. This equipment may be of considerable value in the application of paint generally.

STRUCTURAL STEEL PAINTING

Introduction:

Bridges, gasholders, and other structures constructed of ferrous metal require protection from corrosion, which is the action of the forces of nature to return iron to its stable or combined state as found in the earth's crust. Protection is afforded mainly through the application of protective films of organic coatings, generally referred to as "paint." These protective coatings, therefore, are a means of combating the environmental conditions responsible for corrosion.

The various environments to which iron and steel are exposed in our industrial life may be indicated as follows:

1. Atmospheric
   - (*a*) Urban
   - (*b*) Rural
   - (*c*) Marine
   - (*d*) Industrial
2. Water Immersion
   - (*a*) Salt
   - (*b*) Natural or fresh
   - (*c*) Industrial

It has been estimated that up to 2 per cent of the world's tonnage of iron must be replaced yearly due to corrosion. When it is considered that the world's production of iron and steel is well over several billion tons per year, it is realized that the losses resulting from corrosion run well into many million dollars per year; in fact latest estimates indicate that losses run into a billion dollars per year.

Metal Composition:

The metal most used for structural purposes is iron or the more commonly used form, "steel." Extensive work has been done both here and abroad by the iron and steel companies and their related research organizations to develop steels that would be corrosion resistant. It has led to the development and manufacture of such items as corrosion-resisting or so-called "stainless" steel, "Patino," or copper steel, chromium steel, etc. These alloys are effective for parts exposed to specific corrosive environments and are generally used where the cost of the amount of steel needed is warranted. In the main, however, the large amounts of steel used for structural purposes prohibits the use of the costly special alloyed steels. Work is still going on to develop a low-alloy steel which would compete in price with mild steel and would have similar working properties.

The more resistant low-alloy steels not only have longer life in the bare or uncoated state than structural steel, but when painted show better performance. It seems reasonable to expect that in such alloyed steels the characteristics of the surface would differ from that of mild steel, and they in turn would have their effects on the performance of the protective paint coating.

Many investigators claim that the priming paint coat is usually not in direct contact with the base metal but is rather in contact with an oxide layer, since freshly cleaned metal even if only momentarily in contact with an oxygen-containing environment quickly becomes covered with an oxide layer which may be too thin to be visible. The nature of the oxide layer could be expected to differ with different alloy steels and thus present a different surface condition. In most instances, structural steel shapes are fabricated at red heat which permits the subsequent formation of an opaque surface oxide film usually referred to as mill scale. This film of mill scale will differ in thickness, density, and adherence, depending upon the size and shape of the member as well as on the composition of the steel.

Paints:

Since corrosion is a reaction process between a metal and its environment, either by chemical or electrochemical means, it is reasonable to state that any coating which prevents these reactions can be said to be protective. Paint protects by two main mechanisms: namely,

1. By offering a barrier which mechanically shields the metal from the environment.
2. By inhibiting the corrosive action of any portion of the environment which does permeate the film.

Paints can be formulated which have a high degree of impermeability and will protect for a limited time by physical exclusion of the environment. However, the environment eventually permeates practically all paint coatings used on structural steel and, therefore, the inclusion in the paint of inhibitive type pigments is necessary. It must also be recognized that the best paint from an impermeability standpoint is usually not always the best when wetting, adhesion, and other requirements are considered.

Paints for the protection of metals are designed under two general groups, the primer and the topcoat. The primer or coat next to the metal is formulated to give corrosion inhibitive action as well as good wetting and adhesion. The topcoats are primarily designed to protect the primer, to provide decoration, and to withstand the destructive action of the environment.

The ultimate choice of pigmentation, and particularly the choice of vehicle, is governed by the exposure conditions. One should remember that the protection afforded is provided by a system comprising two or more coats, the compositions of which usually differ. The importance of having each coat perform its intended function and remain compatible with the others should be kept in mind. Therefore, complete knowledge of all factors concerned is necessary before the paint technologist can suggest the most durable system.

The primer coat must protect the metal by inhibiting the corrosive attack, and since, in order to do this, it must stay in contact with the metal, it must possess the additional properties of flexibility and adhesion. The property of inhibitiveness is derived from priming pigments such as red lead, zinc chromate, blue lead, basic lead chromate, zinc dust, and others, or mixture of several of these pigments. The adhesion is primarily a property of the vehicle and its ability to wet or intimately contact the base metal. Linseed oil and other drying oils, alone and together with resins to form varnishes, are the usual vehicles used in priming paints.

Of recent years alkyds, phenolics, coumarone-indenes, chlorinated rubber, and combinations of these have found acceptance in the structural metal protective field. Of all the vehicles, linseed oil has the best ability to penetrate into crevices and residual corrosion products, thus wetting the surface of steel. The synthetic resins possess many special properties which are desirable and by their judicious use, and with due consideration for environment and surface condition of the metal, a satisfactory coating composition can usually be provided. Synthetic resins are often combined with free linseed oil to give faster drying than straight oil and better wetting than the straight synthetic resin type vehicles. Maintained flexibility of a degree which will allow conformity with expansion and contraction changes due to temperature effects is necessary to insure adhesion. Retention of adequate flexibility depends upon proper formulation.

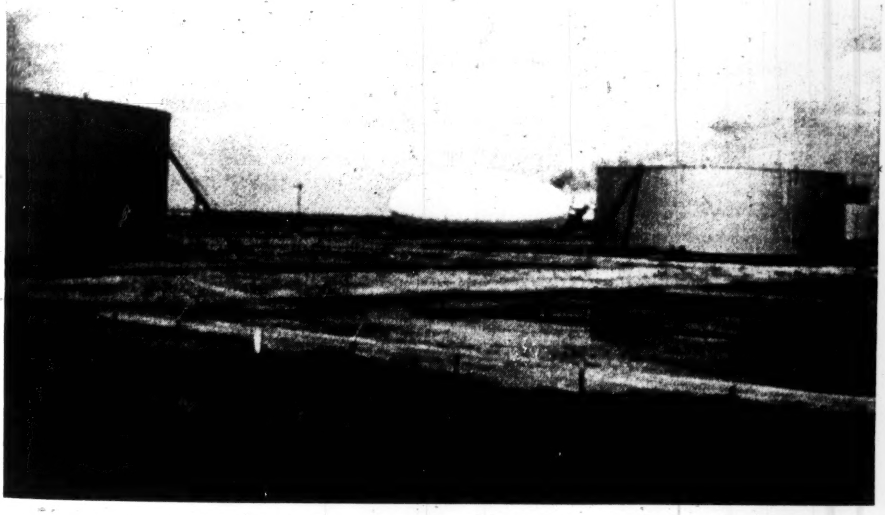

FIG. 35.—Spheroid in Tank Form Painted White to Reduce Evaporation Losses.

Courtesy of E. I. du Pont de Nemours & Co., Inc.

Practically all the recognized corrosion inhibitive type pigments, as already indicated, are basic in nature, and partially react with the acidity of the vehicles commonly used in paint to form compounds, referred to as soaps. The formation of these soaps in the film imparts greater impermeability to the passage of moisture, and these soaps also contribute other properties essential to the inhibition of corrosion.

As mentioned previously, the functions of the topcoats are primarily those of resistance to the action of the environment, which in turn protects the primer coat, and also to provide decoration. In certain instances finish coats are used which have additional special functions such as toxicity to marine organisms in the case of antifouling paints, infrared reflectance properties for camouflage, or temperature control. High infrared reflectances are useful in the petroleum industry in reducing evaporation losses, illustrated in Fig. 35 of a spheroid petroleum storage tank painted white. The formulation of topcoat paints depends upon the environment to which they will be exposed. A wide variety of pigments and synthetic resins makes it possible to formulate paints with varying properties.

Environment and Related Considerations:

Most iron and steel structures are subjected to atmospheric exposure, which generally is a less severe condition than total immersion in river waters, salt water, or environments highly contaminated with chemical fumes. Because atmospheric exposure is less severe, there has been a great tendency to limit surface preparation of the steel to simple scraping followed by wirebrushing. This routine can lead to unsatisfactory results when protective paint is applied, for reasons which will be explained.

Structural steel in normal times may lie exposed in the fabricators' yards and at the erection site sometimes for as much as a year before receiving a protective coat of paint. Depending upon the length of exposure, the mill scale may be 10 to 90 per cent removed and in its place will be rust. This rust is hygroscopic in nature and entraps moisture in the surface over which the paint is applied. It causes subsequent early failure of the film.

Two procedures are most commonly used at present for overcoming this situation. The first is to sandblast or pickle the surface entirely clean of all corrosion products before applying the paint. This is sometimes too expensive or impractical on a completed structure. The other procedure, where surface preparation consists of scraping followed by wirebrushing, as is usually the case with structural steel, is to choose a primer containing a good inhibitive pigment and a vehicle that has a substantial percentage of free linseed oil. Linseed oil has repeatedly proved itself to be an excellent wetter of rusted steel, and a paint containing better than 50 per cent of free linseed oil will penetrate through the rust and crevices and form a good adhesive bond with the base metal. An alternate method is to have the steel receive a coat of paint in the fabricators' shops immediately after being rolled and prior to its exposure. A base of reasonably intact mill scale has generally shown performance second only to completely clean steel.

It should again be emphasized that the safest base over which to apply paint is one which has been completely cleaned of all surface contaminants. If such surface preparation is adhered to and if the paints are properly formulated to withstand the environment, the maximum in serviceability will be realized from the paint system.

Maintenance engineers as well as designing and structural engineers have become more conscious of the part that paint plays in the maintenance of structures. They now view paints as really protective and engineering mediums.

TEXTILE AND FABRIC COATINGS

In the field of textile and fabric coatings only a few interesting uses are given (23).

1. *Oleoresinous Coating:*

A common use of oil-coated fabrics is for wall decoration. Various types of oil varnishes are applied to cloth, which is subsequently printed and then used for wall coverings. This type of cloth wall covering, as well as being decorative, lengthens the life of the interior finish by preventing plaster cracks. It also aids in insulation.

Another protective finish, consisting primarily of oil, is applied to cloth used as a padding material or welt between the body of an automobile and fenders. This type of application has been used extensively and very effectively. Acting as a cushion, it absorbs considerable shock and noise.

2. *Cellulose:*

Among the more recent uses of protective fabric coatings is their use in planes. Although for some time fabric has been applied to various plane assemblies and then sprayed with a cellulosic lacquer, the use of a precoated fabric is relatively new. For this purpose the cloth is coated with the cellulosic coating and, after smoothing, is applied to the plane. The application of this type of precoated fabric eliminates considerable sanding and a number of spray coats. Saving is considerable.

3. *Vinyls:*

The vinyls have one advantage over many other synthetic coatings in that they can readily be fireproofed. Advantage is taken of this feature in making bomber insulation materials. The aviation industry also makes use of vinyl coatings on glass cloth in a heating tube which is used to heat engines and cabins in planes. (See Fig. 36.)

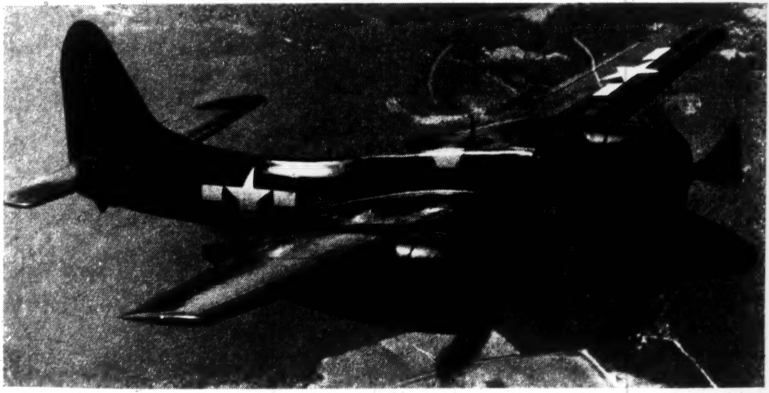

FIG. 36.—"Tigercat" Airplane with Smooth Exterior Finish.

Official Photograph U. S. Navy

Vinyl and synthetic rubbers are now being used as coatings for glass cloth which is used for construction of hangar doors and water tanks.

4. *Synthetic Rubber:*

Cloth coated with various synthetic adhesive materials is used as a backing for veneer to give decorative wall finishes. The veneer is laminated to the cloth, and the cloth gives sufficient strength for ease of handling. Synthetic rubbers of all types are used in life rafts and flotation gear.

TRANSPORTATION PAINTS

Naval Aircraft Coatings:

Introduction.—There is discussed in this section the rôle of organic coatings in naval aircraft engineering since 1939. The well known motto, "Save the Surface and You Save All," as applied to Naval aircraft, has been found to be strictly apropos. The advances of modern technology in the design and construction of Naval aircraft and its component parts would be nullified to a great extent if the deleterious effects of the elements and the severe operating conditions encountered in service were not offset by means of organic protective coatings. The finest mechanism or even the entire flight article can be destroyed by the destructive forces of the environment on vulnerable aircraft metal, fabric, and wood surfaces. The small margin dividing victory and defeat in combat may often be determined by the thin film of protection afforded by organic coatings.

Painting of exterior and interior surfaces of aircraft structures and equipment provides one, two, or all three of the following functions:

(*a*) Corrosion protection,
(*b*) Aerodynamic smoothing properties,
(*c*) Color requirements.

The Navy has found paint to be indispensable in both war and peace in maintaining and protecting aircraft. Naval aircraft operate under extremes of weather, alternating from the intense cold of high altitudes and arctic conditions to the high temperatures which prevail at sea level in deserts and in the tropics. The safety of the airplane and its flight personnel may be dependent upon the proper functioning of the paint to provide adequate protection against such conditions. Long periods of inactivity also result in aggravated corrosive attacks. Proper functioning of delicate electronic equipment, vital engine parts, airfoil mechanisms, etc., may be disrupted by lack of protective coatings. These problems were, in the main, solved by means of paint. Increased speed and range were attained by use of proper surface coating and finishing. Problems of camouflage and identification were solved through the medium of paint. The preservation of parts, equipment, and aircraft, both in transit and in storage at depots, was effected by means of organic coatings.

The primers, varnishes, lacquers, enamels, dopes, and specialty finishes employed by the Navy for aircraft are the result of the utilization of the best possible ingredients obtainable, the optimum physical requirements being necessitated by the adverse operating conditions. The finishes must be resistant to corrosive media and abrasion, maintain maximum elasticity and adhesion, and provide aerodynamic smoothness. Advances in processing and improvements in materials are constantly being incorporated into Naval Aircraft finishes.

Navy finishing schedules include the following materials:

1. Primer, AN-TT-P-656, is a modified alkyd, phenolic combination vehicle containing zinc chromate pigment as the anticorrosive ingredient.
2. Lacquers used are essentially alkyd-modified nitrocellulose and are represented by the glossy lacquer AN-L-29 and camouflage lacquer AN-L-21. The glossy lacquer is softer than lacquers used in the automotive field, will not sand or polish as easily, but is more flexible. Camouflage lacquer has a dull matte finish.

3. Enamels used are alkyd formulations and are represented by the glossy enamel, AN-E-3, and camouflage enamel, AN-E-7. These enamels are more durable than the lacquer, especially on the hulls of flying boats, but the slower dry presented an operational disadvantage. This disadvantage was largely overcome during the last stages of the war by improvements in resins which resulted in a camouflage enamel that dried much more quickly.
4. Varnishes are represented by the glyceryl phthalate spar AM-TT-V-116, and phenol-formaldehyde spar, AN-V-26. These varnishes are used chiefly to protect wood and plywood surfaces of aircraft.
5. Dopes are used to finish fabric surfaces on aircraft. The principal dope in use is a cellulose acetate butyrate type, AN-D-1 (clear) and AN-D-2 (glossy), pigmented, and AN-D-3 (camouflage), pigmented.

Corrosion Prevention.—Naval aircraft are constructed from a number of types of metals and alloys, the choice depending upon the function to be fulfilled and the physical properties required of the part. The major portion of the aircraft structure is fabricated from aluminum alloys because of strength and weight considerations. Sheets for wing and fuselage are almost exclusively fabricated from aluminum-covered aluminum alloy to increase corrosion resistance. Ar-

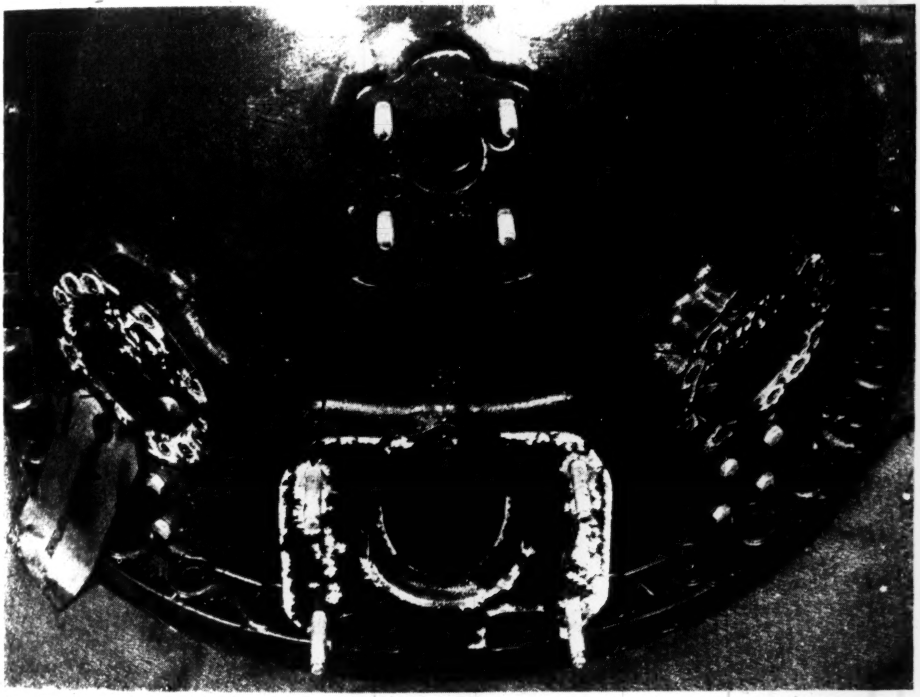

FIG. 37.—Corrosion of Magnesium Nose Section of Aircraft Engine Due to Improper Priming of Faying Surfaces and Improper Insulation of Dissimilar Metal Contacts, That Is, the Cadmium-Plated Studs and Inserts.

Official Photograph U. S. Navy

resting hooks, armor plate, bolts, fittings such as turnbuckles, landing gear, antifriction bearings, control cables, engine parts, etc., are constructed of steel. Landing wheels, engine cases, etc., are made of magnesium. Steel and magnesium metals are readily corroded and require exceptional protection to prevent deterioration of the parts. In addition, the presence of these dissimilar metals in contact with each other or of steel or copper alloys in contact with aluminum causes an aggravated corrosion condition since the corrosive effect due to chemical action of the surrounding environment is increased by the galvanic action of the dissimilar metal contact. Examples of corrosive conditions likely to be encountered in service and requiring precautionary protection measures are severe corrosion of interior surfaces of wings, etc., due to condensation of moisture, pitting and corrosion due to salt spray and coral dust, galvanic corrosion of aluminum alloy in contact with poorly plated steel fittings, and others too numerous to list here.

The Navy's Bureau of Aeronautics has expended considerable effort in the basic design of aircraft structures, altering the types of metals employed in order to increase the resistance to corrosion from galvanic and chemical actions, wherever possible. However, because of unique special requirements, the optimum corrosion resistance could not always be attained. Since structural strength could not be compromised in aircraft construction, corrosion resistance was, of necessity, subordinated to strength considerations, and consequently organic coatings were called upon to achieve the objective. (See Fig. 37.)

In the manufacture and maintenance of Naval aircraft, the interior and exterior of such parts as wings, fuselages, cowling, struts, empennage, and floats are treated in strict conformance with NavAer Specification SR-15. In addition, the finish of propellers, power plants, and engines is strictly controlled. Such standard accessories as electric motors, generators, instruments, switches, rheostats, relays, hydraulic valves, pumps, and similar parts must have a finish affording satisfactory protection against corrosion.

In the manufacture of airplanes, aluminum and magnesium parts are given a chemical surface treatment in accordance with Specification NavAer SR-147, followed by a zinc chromate primer. These chemically surface treated and primed parts then become the building materials for making larger assemblies for fabrication of combat airplanes. (Certain alloys for trainers and transports remain unpainted because frequent periodic maintenance of the exterior of these types of planes is available. This was not the case with combat planes during the war.) Faying surfaces, seams, butt joints, and cut edges of 25 aluminum, and 3S, 52S, 53S, 61S, 17S, 24S, and 75S aluminum alloys and their aluminum-clad counterparts, as well as all contacts of dissimilar metals, are protected with zinc chromate primer or zinc chromate sealing compound. When water enters a joint containing zinc chromate primer or sealing compound, some of the zinc yellow pigment dissolves and an inhibiting action takes place which tends to suppress the corrosive effect of foreign matter such as water, salts, and organic acids, thus maintaining the integrity of the metal. As a result of strict adherence to Specification SR-15, both by Naval aircraft manufacturers and Naval maintenance, problems of corrosion on Naval aircraft, both galvanic and chemical, were eliminated in combat localities. Where other than minor deviations from the specification had been practiced, corrosion was usually found to result. (See Fig. 38.)

The primary function of chemical surface treatment of metals is to improve the adhesion between the metal substrate and the primer. Although some corrosion resistance is contributed by chemical surface treatment, this function is subordinate to its primary rôle as a bonding agent for paint. Electroplated coatings such as cadmium or zinc plating in themselves have been found inadequate to prevent corrosion over long periods. These parts on aircraft must, therefore, be painted.

AN-TT-P-656 zinc chromate primer is the basis of all anticorrosive organic finishing systems used on aircraft metal surfaces by Naval aircraft establishments and the Naval aircraft manufacturing industry. Furthermore, it serves as a base for subsequent topcoats. Since the value of any finishing system is no better than the adhesion attained, the importance of the primer cannot be overemphasized. Consequently, the Navy has expended considerable effort in improving the composition of the primer and methods of application.

Because of the extreme demands on the primer for maximum anticorrosive and adhesion properties, these physical characteristics were developed to the greatest degree and the weathering characteristics considered secondary. To provide adequate durability and aging properties (besides aerodynamic smoothness, camouflage, and identification), topcoats are employed. One coat of enamel or varnish is specified by Specification SR-15. In lieu of this coating, two coats of lacquer may be used. One coat of lacquer may be used, but because of the low solids content, a film too thin to withstand weather exposure will be deposited. Navy BuAer varnishes and enamels possess excellent durability characteristics. Lacquers are used, particularly in overhaul activities, because of their rapid drying time with resultant speed and economy of operation, particularly during overhaul, touch-up, and changes in color schemes.

A corrosive condition which presented

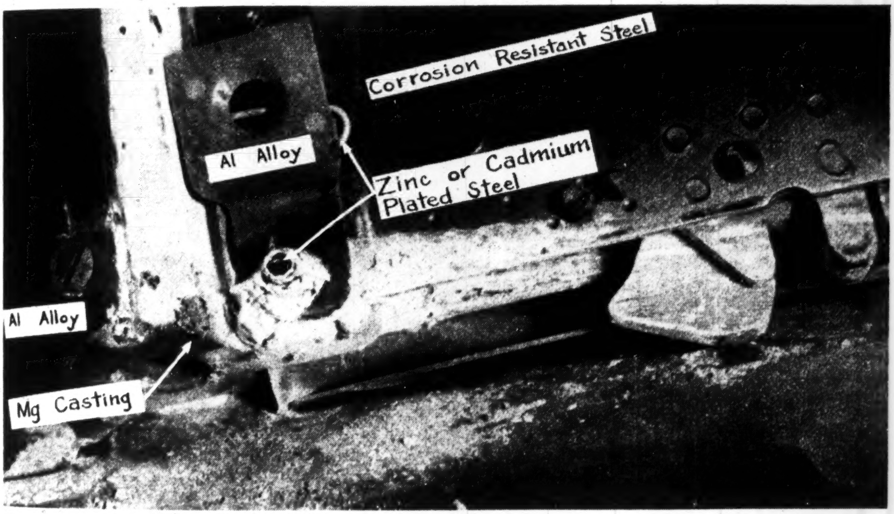

FIG. 38.—Corrosion of Uncoated External Bomb Rack Due to Improper Insulation of Dissimilar Metal Contacts.

Official Photograph U. S. Navy

difficulties was the rusting of interior surfaces of steel dropable fuel tanks. These tanks were originally designed for one mission and were to be discarded before the aircraft returned. However, the tanks were returned and reused on numerous occasions. With the close of the war, moreover, the tanks were no longer classified as expendable because of their cost. Since it was found impossible to keep a full supply of gasoline in the tanks at all times, and since ferrous surfaces, particularly in the clean condition resulting from contact with the gasoline, tend to corrode rapidly, rust formation was encountered on the interior of the tanks. An aromatic fuel-resistant lining material was developed which is unaffected by gasoline containing as much as 40 per cent aromatics and satisfactorily prevents interior surface corrosion. This material is of the air-drying type, NavAer Specification M-769.

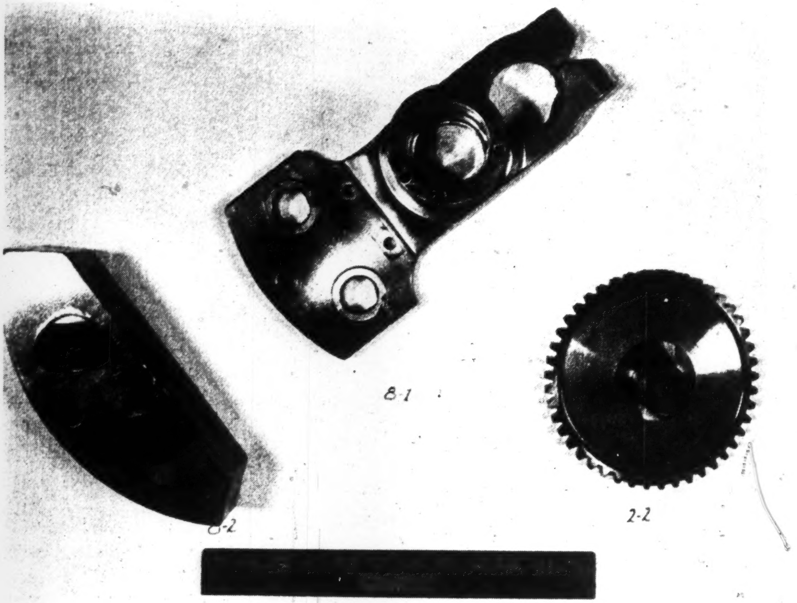

FIG. 39.—Aircraft Engine Parts Coated with Resin.

Official Photograph U. S. Navy

Engines which had been received in operating theaters of war in good condition as a result of preservation with moisture barrier envelopes, preservative compounds, and dehydrating agents were frequently found to have undergone serious corrosion on the internal steel parts when the engines were installed in airplanes. Corrosion of the engine parts also occurred during operation of the planes. As a result of extensive investigations a suitable material and application procedure covered by Specifications AN-C-148 and AN-C-158 were devised to combat this corrosion, to provide additional protection for spare or disassembled parts either in storage or during overhaul, and to reduce labor necessitated during the subsequent overhaul. (See Fig. 39.) The material is a 100 per cent phenol-formaldehyde resin and is sprayed so as to obtain a final film thickness of approximately $\frac{1}{2}$ mil. The film is baked at 300 F. to form a coating resistant to gasoline, hot lubricating oils, greases, and similar compounds, and it eliminates corrosion. All the interior steel parts of scores of engines were resin coated and found to be in excellent condition after hundreds of hours of operating service. The resin is applied to master rods, articulating rods, crankshaft assemblies, springs, cams, cam-tracks, gears, etc. Although this development came to fruition in the later stages of the war, it is believed that it will find increased use during peace not only from the standpoint of minimizing maintenance of aircraft engines and saving of expensive engine parts from discard, but also from the standpoint of the preservation of the parts in storage.

Because of the elevated temperatures existing at various locations on the airplane, serious corrosive effects have resulted. Engine exhaust gases and gun blast gases were found to have corroded gun blast tubes, areas adjacent to wing guns, fuselage areas, exhaust collector rings, etc., as a result of the extreme heat and the corrosive chemicals present. The increased use of jet-propelled aircraft will aggravate high-temperature corrosion problems in the future. A partial solution to the problem is the use of AN-TT-E-501 baked enamel or aluminized varnish.

The susceptibility of magnesium to corrosion necessitates the use of protective coatings. In aircraft magnesium landing wheels the corrosive action was found to have been intensified by the failure of cadmium plating on steel inserts, resulting in dissimilar metal contacts. Moreover, owing to excessive use of brakes, high temperatures were produced which resulted in partial decomposition of the protective coating and accelerated corrosion of the magnesium wheel. The finishing scheme was accordingly improved to eliminate these undesirable effects. The cadmium-plated inserts were laid in zinc chromate primer, and after this treatment, the entire wheel was coated with zinc chromate primer and then finished with two coats of aluminized lacquer. Data resulting from recently completed experiments indicate that the finishing scheme can be further improved by substituting enamel for the aluminized lacquer on magnesium.

With the advent of war and shipment and storage of aircraft and aircraft components and accessories to various parts of the world, exposure to corrosive conditions increased enormously. The high humidity, entrapped moisture, bacteria, mold, insects, and other adverse agencies encountered at numerous supply depots played havoc with all types of equipment and materials. Organic protective coatings, both permanent and strippable, and oil type preservative compounds were employed as approved in AN-P-13, the Preservation and Packaging Specification, to overcome these conditions.

Aerodynamic Smoothness.—The Navy employed camouflage finish on aircraft in the early part of the war. With the improvement of radar and the swing over of the initiative to the Allies, however, it became increasingly evident that the passive defense value of camouflage finishes might no longer be necessary and the trend to glossy finish was under way. Glossy finishes weathered better than camouflage, as would be expected. In fact, the better appearance value of the glossy finish was one of the factors considered in the later change from camouflage. It was proved in service that a pilot had more confidence in a shiny than a dull airplane. A controversy existed regarding the effect of surface finish upon aerodynamic efficiency, and the importance of this problem was emphasized with the development of higher and higher performance aircraft. Flight tests under accurately controlled flight and power conditions were accordingly conducted by the Bureau of Aeronautics after each individual phase of surface condition of a fighter airplane, emphasis being placed upon a sufficient number of flights to establish definitely the reproducibility of performance data in order to establish the dependability of the test results. Full advantage was taken of all available technical and engineering knowledge and devices, such as, profilometer, torque-meter, electric-eye, pacing aircraft, master instruments, aircraft scales, etc. Flight tests were made with six different surface finish conditions, and each phase of surface finish was accurately defined in terms of brake horsepower *versus* true air speed with an established accuracy of ±1 mile per hour. The six different surface finish conditions employed were:

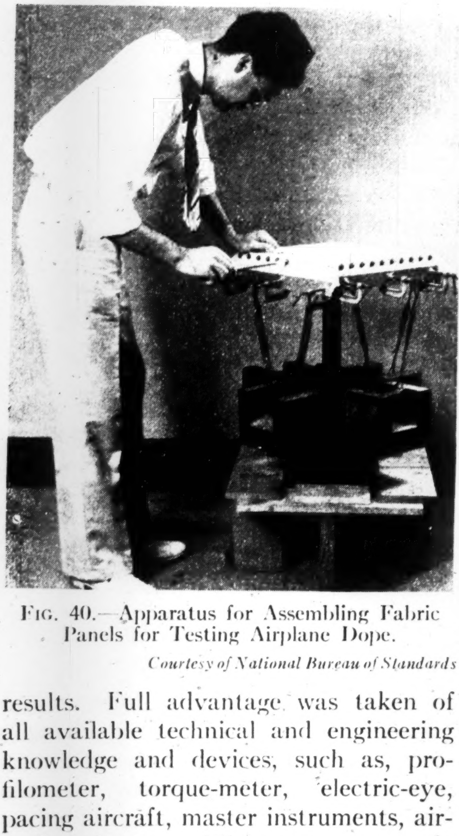

FIG. 40.—Apparatus for Assembling Fabric Panels for Testing Airplane Dope.

Courtesy of National Bureau of Standards

Condition 1—Representative condition of a fighter airplane after 25 hr. operational service.

Condition 2—Painted but not smoothed, no fairing of poorly fitted joints of skin plates.

Condition 3—Paint smoothed, no fairing of poorly fitted joints of skin plates.

Condition 4—Painted but not smoothed, airplane thoroughly faired before being painted.

Condition 5—Paint smoothed, airplane thoroughly faired before being painted. (This represents the closest approach to a true aerodynamically smooth surface.)

Condition 6—Bare airplane, unpainted.

(*a*) Seam joints and screw heads contained fairing compound.

(*b*) Seam joints and screw heads stripped of all fairing compound.

An analysis of the test results substantiated the following conclusions:

1. Production paint coatings could be applied to aircraft in a practicable manner without resultant loss of speed.

2. The direct adhesion of aircraft finishes to aircraft surfaces was inherently acceptable.

3. Surface finishes could be no more durable than the undercoats over which they were applied.

4. All available fairing putties failed and introduced an increment of drag much greater than any possible gain.

5. The application of the best practicable smooth finish at the time of manufacture, and, if it was retained in service by reasonable care and maintenance, it would enhance the maximum performance of a 400-mph. fighter airplane to the extent of at least 13 mph., which is equivalent to 140 hp., or a proportional increase in range at a given speed with a given gasoline supply. The effect of finish on speed becomes more pronounced as the speed increases.

The chance of an airplane coming

back from a mission might depend upon these extra miles per hour in a dogfight or, in case of a bomber returning home from a mission, the supply of gasoline might be just sufficient to bring the plane home, since smooth paint finish increases the range with a given gasoline supply.

A color movie with sound, entitled "Painting Naval Aircraft," was released during the war, and a first-hand study was also made of the painting facilities, personnel, and local conditions at various Navy aircraft contractors' and subcontractors' plants, in order to assist in the engineering of aircraft paints at each individual locality. As a result of all this groundwork, a specification, NavAer SR-156, entitled "Finishes; Aircraft, Application and Treatment for Producing Aerodynamically Smooth Surfaces," was issued, in which were presented the proper techniques for cleaning prior to priming, cleaning prior to application of topcoats, application of primer, application of topcoats, tack-ragging, spray-gun handling technique, adjustment of gun nozzle and pressures, proper hose sizes, scaffolding, use of sanding, waxing, and cutting equipment, and proper spray-room air conditions. Standard panels showing acceptable smoothness were prepared for distribution for judging the results obtained by spray painters. It is reasonable to state that the proper application of paint to an airplane exterior surface will yield an aerodynamically smooth paint finish. This, of course, presupposes that clean aerodynamic design and careful production, which insures aerodynamically smooth contours, have been built into the airplane by the contractor. No amount of painting will cover up gross manufacturing deficiencies.

Fabric Protection.—Although the trend in modern aircraft is toward all-metal construction, fabric is used extensively on control surfaces. The advantage derived from the use of fabrics results from the fact that fabric is extremely light in weight and consequently weight distribution can be improved. In addition, the material is readily removed for inspection. However, fabric possesses a number of disadvantages which must be overcome before it can be used on aircraft, for example, poor weather resistance, high moisture permeability, low fungus resistance, excessive slackness, insufficient strength, and excessive surface roughness. These problems were solved by the use of lacquer dopes. Airplane dopes are applied to fabric aircraft surfaces (see Fig. 40) to render them taut and impermeable to air and water, provide protection against weather and mechanical damage, maintain flexibility and durability, and provide additional strength in order to support the weight and air pressures encountered by the fabric surface during flight. The dope must provide sufficient tautness, since deficiency in this property will result in a decrease in load-carrying

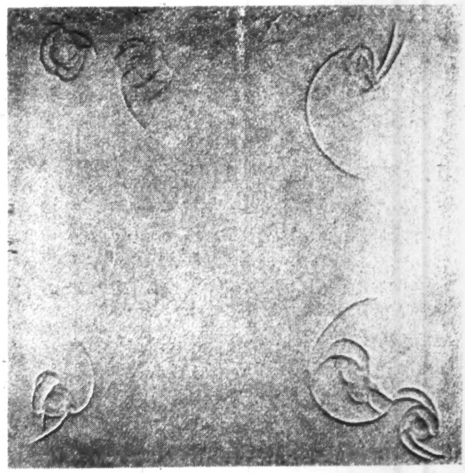

FIG. 41.—Doped Fabric Which Has Ringwormed Because of Brittleness of Dope Film.

Courtesy of National Bureau of Standards capacity and flight control of the plane. Smoothness is here also essential since the greater the speed of the plane, the greater the effect of surface irregularities on the aerodynamic efficiency of the plane.

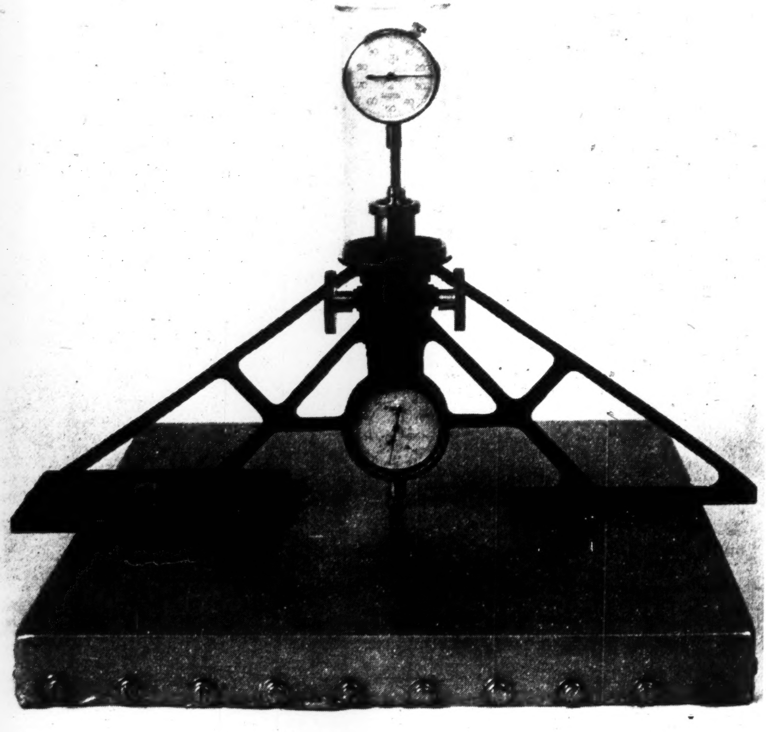

FIG. 42.—Kline-Schiefer Tautness Meter in Position for Making Measurements.

Courtesy of National Bureau of Standards

Originally, the Navy used cellulose nitrate dopes and cellulose acetate dopes. Cellulose nitrate was in disfavor because of its flammability. Cellulose acetate doped fabric, however, although eliminating the fire hazard, was prone to lose tautness when exposed to moist atmospheres. As a result of developmental work, a lacquer dope, AN-D-1, based on cellulose acetate butyrate was adopted, which material develops satisfactory tautness, is nonflammable, durable, and impermeable to moisture. It maintains satisfactory flexibility on aging and outdoor exposure. If lacquer dope is brittle, the coated fabric becomes ringwormed as shown in Fig. 41. The dope is formulated to develop shrinkage forces great enough to tauten the fabric (see Fig. 42) but not sufficient to warp the frame to which the fabric is attached. The formulation is such as to provide quick drying, have no tendency to trap air bubbles or foam, and to produce smooth surfaces and adhere adequately to the fabric. Exposure racks for testing of doped fabrics are shown in Fig. 43.

Since fabric, even though well coated with dope, is highly susceptible to fungicidal attack, investigations were conducted to combat any resultant fabric deterioration. Excellent results were obtained by incorporating salicylanilide into the first coat of dope. However, marked yellowing of white dope topcoats resulted. Further investigations led to the use of zinc dimethyl-dithio carbamate which was more potent and avoided the discoloring effect of salicylanilide. Pigmented dopes are formulated by the addition of pigments to the cellulose acetate butyrate dopes for use as topcoats for the purpose of increasing the durability of the fabric finishing system, since exposure of the clear dope film to the sunlight would result in a greatly increased rate of film decomposition.

A most important item in the preparation of satisfactory fabric finishing is the method of application. Although the composition and performance of the dopes are rigidly prescribed, the application technique is of such importance that deviations therefrom would yield poor results. Accordingly, NavAer Specification SR-70 was promulgated to outline an exact procedure to insure proper application. All fabrics are doped in air-conditioned rooms at approximately 50 per cent relative humidity and 70 to 80 F. to prevent blushing. The first two coats of clear dope are brushed on in order to obtain thorough penetration of the fabric. Sanding between coats is necessary.

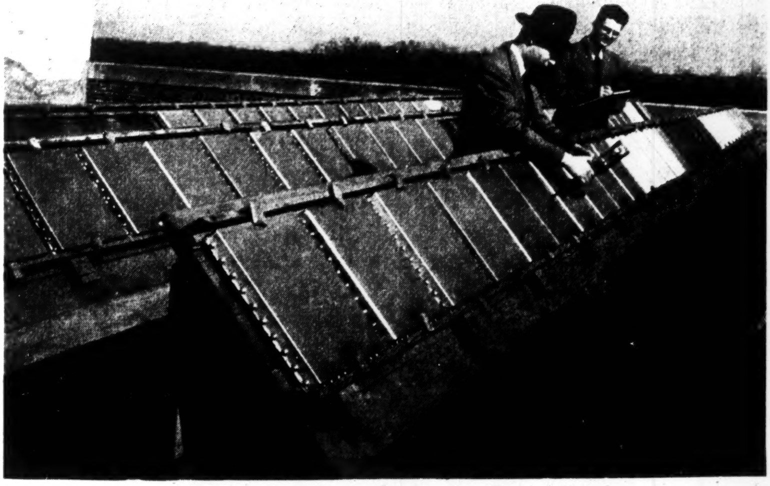
FIG. 43.—Exposure Racks for Testing Doped Fabrics.

Courtesy of National Bureau of Standards

Two additional coats of clear dope and finally two coats of pigmented cellulose acetate butyrate dope, AN-D-2 (gloss) or AN-D-3 (camouflage), are sprayed on. The total weight of applied material is specified as 4.0 to 4.5 oz. of dope film per square yard of cotton fabric.

Indications are that airplane fabric will not be used for airplanes having speeds much in excess of 400 mph.

Wood Protection.—Although the use of wood on aircraft is limited, some trainers during the war employed the wood and fabric type of construction. An early attempt to use direct-on-wood paint finishes failed because of deterioration of the finish. Painted fabric-on-wood remained as the standard exterior wood finish scheme for this type of training plane. In this case, the surface of the wood is cleaned of any glue extrusions or smears, by sandpapering, and the surface is wiped with tack rags and then with solvent. The wood is sealed with three coats of thinned varnish, AN-TT-V-116 or AN-V-26, the first coat being brushed on. Two coats of clear nitrate dope, Specification AN-TT-D-514 are applied and the fabric cemented down. Three coats of clear nitrate dope are then applied on top of the fabric, followed by two coats of pigmented nitrate dope, Specification AN-TT-D-554. It was found that nitrate dope is a better cement for fabric than cellulose acetate butyrate dope, and, because of this improved adhesion, the Navy considered it advisable to employ a complete nitrate dope scheme in this case. Cellulose acetate butyrate dope continued, however, to be the standard Navy dope for fabric surfaces of all combat type aircraft.

With the advent of the sandwich type of aircraft construction material, consisting of a fiberglass or aluminum layer cemented on each side of balsa wood, the complexity of the finishing procedure increased. The aluminum materials cannot be given an anodized surface treatment such as conventional aluminum sheet would receive, since the bath would attack the layer of wood. A phosphoric acid brush-on treatment is employed on the outside of the aluminum layers to secure paint adhesion, followed by application of the standard paint scheme. The edges of the wood are sealed with two coats of aluminized AN-V-26 varnish (16 oz. aluminum per gallon of varnish). In the case of fiberglass sandwich material, the fiberglass receives an application of seam compound to fill up the voids, and the entire unit is coated with two coats of aluminized AN-V-26 varnish. A polyester resin is applied to the fiberglass and cured during construction of the material to seal it and to prevent the ingress of moisture. Because of the poor weathering properties of the polyester resin, additional topcoat protection is required. Hence the necessity for the aluminized varnish topcoats mentioned above.

Color.—During the early stages of the war, the camouflage scheme chosen for Naval aircraft was the concealment type in preference to the "razzle-dazzle" or deception type. Permanent coatings were chosen rather than the application of a removable type of camouflage on top of a basic gloss paint scheme. Initially, the color scheme was often changed in a constant endeavor to adjust the color of the plane to match the background against which it would be viewed to make for maximum concealment, with the result that new colors in the finishes were constantly being developed. Later, a standard camouflage color scheme was selected and universally applied to all combat Naval aircraft. During the final stages of the war, camouflage was retained only on flying boats and on a few other aircraft, while fighters and attack planes were given a glossy finish. One camouflage scheme used during the war, the BuAer Anti-Submarine Warfare Color Scheme, which was applied to aircraft engaged mainly in this type of operation, is worthy of mention. This scheme furnished the Navy airplane an extra minute of concealment before being spotted by an enemy, which spelled the difference between losing contact and being able to destroy the submarine before it could crash-dive to safety. With the improvement of radar, however, the situation was changed.

The new national aircraft insignia, with its panels on each side of the white circle, made it possible to distinguish friendly airplanes at a greater distance than did the old round insignia, since the new insignia appears like a long dash when viewed at a great distance. Enemy insignia appear like a dot when viewed at the same distance.

Quality Control of Paint.—Organic protective coatings are called upon to protect not only aircraft but with it the lives of highly trained and courageous personnel. In addition, the money value of aircraft and aircraft components is enormous, when compared with the cost of the applied paint, whose function it is to protect these items. Consequently, the need for exercising rigid quality control of the paint materials applied by prime contractors, subcontractors, and the Naval Air Establishment has always been recognized in the Bureau of Aeronautics. Every batch of paint made by the paint manufacturer for use on Naval aircraft is sampled, tested, and passed upon in accordance with the applicable material specification. Samples which fail to pass the laboratory tests are rejected, or, when the variation is minor, use of the paint is authorized under very limited and well-controlled conditions.

Thousands of panels of aluminum, steel, fabric, and wood were exposed at Philadelphia, Cape May, Chapman Field, Fla., and Tahiti Beach, Fla., to determine the outdoor weathering properties of aircraft paints. No paint supplier received permanent approval to vend his submitted materials until satisfactory completion of weather exposure tests.

This elaborate control system yielded dividends in the appearance, performance, and reduced maintenance of Naval aircraft. However, the weather exposure facilities of the Navy's Bureau of Aeronautics are too limited and a new subtropical exposure station is under construction on Fisher's Island in Biscayne Bay, adjacent to Miami, Fla. This tract is at approximately 26 deg. north latitude. One large tidewater rack and ten smaller land exposure racks will be constructed there, with capacity for thousands of panels of Navy aeronautical and Army-Navy aeronautical finishes. No less important is the rôle these racks are destined to play in the research and development program of BuAer for the improvement of aircraft finish. The racks will be of concrete and monel metal construction and should last for many decades. It is still realized, however, that even these outdoor exposure tests do not yield the final evaluation of aircraft paints. None of these tests takes into account the effects of vibration, "oil-canning," and flexure, such as that which takes place in wings during flight. However, for all practical purposes, the existing paint testing methods will predict the relative value of paints compared with those which have been tested in this manner in the past and whose performance history in actual aircraft use is already known.

The shortages of tung oil and toluene and the recurring shortage of phenolics and alkyds during the war led to a considerable amount of investigation and testing work to keep the specifications for the paint products in line with available material. It was necessary to replace the tung oil in primer with linseed, dehydrated castor, soybean, or other available drying oils, without any sacrifice in speed of drying, water resistance, or topcoat recoating properties. The AN-TT-P-656 primer developed during this period of shortages actually proved to be more flexible and durable than the previous AN-TT-P-656 primer, which contained a greater amount of tung oil.

The shortage of high aromatic solvents necessitated the testing of substitute thinners for specification paint materials, and the specifications were constantly revised during the war to parallel the changing availability of thinners. As a result, specification AN-TT-T-256 for thinner was amended six times during the war.

Specialty Paints.—A number of developments on protective coatings by the Bureau of Aeronautics that have not been noted are worthy of mention. A few of these are outlined briefly below.

Transports and some trainers are usually constructed of aluminum-covered aluminum alloys for the exterior skin. The practice of waxing the surface to maintain suitable appearance is unsatisfactory owing to the short life of the wax coatings which would necessitate excessive overhaul space and a large personnel for maintenance purposes. A clear, water-resistant, air-drying finish which forms a thin protective film was developed. The product, NavAer Specification L-22, is chiefly a methyl methacrylate resin composition which has excellent resistance to adverse environmental conditions. It is applied in a $\frac{1}{2}$-mil thickness over solvent-washed aluminum-covered aluminum alloy and requires a minimum of maintenance.

Rubber tires of aircraft were found to be adversely affected by exposure to sunlight, moisture, etc. To combat this condition, a flexible rubber paint, M-529, was developed which successfully protected the tires and notably increased their durability.

The shock and abrading action occurring when persons walked on a plane impaired the paint coating and surface of the plane. To prevent this, and also to reduce personnel hazards due to slippery surfaces, walkways are employed. A brush-on type of walkway paint was developed for use on aircraft wingwalks. Two types are used:

1. Type 1—Smooth, for use along leading edges and adjacent surfaces of high-speed aircraft where roughness is undesirable because of its effect on aerodynamic properties. This resulted in presenting a smoother surface to the air in the forward part of the airplane, where aerodynamic efficiency is of greatest importance.
2. Type 2—Rough, for use along trailing edges and adjacent surfaces where maximum nonslip qualities are essential. In the after portion of the wingwalks, conventional rough walkway material also continued to be employed.

Future Protective Coating Requirements and Research.—As a result of wartime experiences in the Navy's Bureau of Aeronautics with aircraft finishes and related problems and the research and developmental work conducted to date, as well as the anticipated problems evolving from the development of planes with supersonic speeds, jet-propelled mechanisms, etc., a research program is under way to supply adequate surface protection for aircraft against the markedly increased severity of anticipated service conditions.

Automotive Paints:

Automotive protective coatings, paints, enamels, and lacquers only in a few instances increase the durability of any mechanical parts of the automobile. Morrison of Chrysler Corporation calls attention to one case where a paint applied to a mechanical part increased its operating efficiency. Reference is made to a lacquer crankcase sealer used to seal up any sand particles that might be embedded in the casting. In the operation of the car, the flow of oil would loosen these particles if they were not sealed in and this would cause eventual plugging of the oil line.

It should be borne in mind, however, that paints, enamels, lacquers, and varnishes as applied in the automotive field are in themselves engineering materials because they require the same ingenuity in formulation, meeting of operating conditions, proper selection of color combinations, durability to outside exposure, etc. For this reason, a thorough technical appreciation of the methods of applications of the protective coatings as well as material standards are absolutely essential.

Morrison's experience takes us back some thirty years, when most of the protective coatings used on automobiles were varnishes and in some cases black high baking japans. Little protection was obtained from the varnishes. As a rule these coatings "checked" inside of a year and required refinishing. Also very little protection was obtained against rusting of the metal parts. Better success was obtained from high baking japans where steel parts, especially automobile bodies and fenders, were protected for as long a period as ten years. Unfortunately this type of material permitted the use of only one color—black. Inasmuch as the public demanded colors, this material was gradually discarded. Extreme care should be exercised in the selection of coatings for the purposes intended with respect to both durability and economy. For example, one would not think of using the same type of enamel or lacquer on the body and sheet metal parts of the car as were applied to the chassis frame. Not that they would be inferior in any way; in fact they would do a better job than the paints ordinarily used for this purpose, which are usually of the cheap asphaltic type: these cheap paints fulfill the performance requirements because the coated parts are invisible.

Protective coatings are applied to automobiles, trucks, and other automotive items for protective and decorative purposes. Protection means protection against corrosion of exposed metal parts due to atmospheric conditions such as salt air along the sea coast and salt used in winter on the streets, as well as oxidation of the metal itself. In the northern cities a great amount of salt is spread on the streets in the winter. For example, in 1942–43 the city of Detroit spread 22,872,000 lb. of salt. This salt contributes definitely to corrosion. Protection also means protection against high humidity conditions which cause blistering of the paint films and eventually rusting of the metal; protection against changes in temperature in going from warm to cold which frequently causes "checking" of the paint film and eventually loss of protection against the metal; protection against water, abrasion of road particles encountered in driving, perspiration (as in the case of the steering wheel), and actual preservation of the wood in such wood parts as truck stakes and platforms.

Many cases of "paint failure" in the past were actually due to rusting from the inside in spite of the protection of the exterior coatings. The automotive industry is becoming increasingly conscious of the necessity for painting the interior surfaces of metal to prevent the corrosion by water which may get into the body through rain, washing, or condensation.

It might be well to mention at this point that the protective efficiency of enamels, lacquers, and paints has been greatly enhanced in the past ten years by preliminary treatment of the body and sheet metal parts before paint application. These treatments are of the inorganic materials, but they play a very important part in combination with the finishes used to lengthen the life of metal parts going into the automobile or truck. Reference is made to such treatments as bonderizing, parkerizing, and deoxidine coatings. In combination with finishes, these coats prevent the spreading of rust when the paint film becomes marred or scratched to such an extent that the bare metal is exposed. In such cases salt spray corrosion resistance of enamels and lacquers has been increased as much as ten times, and at the same time better adhesion of the protective coats is obtained.

Most automobiles are finished in one of two major protective coating systems. On the one hand is the lacquer system. Here the first coat is an oil-base primer, baked dry. This provides for the maximum bond between succeeding coats and the metal. Then a highly pigmented oil-base surfacer is baked on to provide a smooth surface over which is applied the decorative as well as protective lacquers. The extremely fast drying of the finish in this system permits greater brilliance of metallic appearance and lends itself particularly to the two-tone decorative schemes. On the other hand, some car makers employ an enamel system. Here the first coat applied to the metal is a synthetic resin type combination primer and surfacer which, when baked dry, supplies both the bond to the metal and the smooth surface for the color coats. The synthetic enamel finish is then applied and baked dry in one or more coats.

As to the decorative effect of protective organic coatings, appearance of the car has as much to do with sales appeal as anything else. As a matter of fact, the public has become color conscious and is greatly influenced by color combinations. One might well ask how appealing would an uncoated and corroded car be to anyone, particularly to the ladies.

Great strides were made before the war in increasing the durability of paint films and thus increasing the life of the metal parts. Unfortunately, the war brought this progress to a standstill because of the scarcity of the materials involved which were necessary in producing improved coatings. For example, the newly developed melamine resin has done much to harden enamel finishes and permit faster production schedules, and produce smooth finishes, pleasing to the eye. Now with the restrictions on most materials lifted, the paint companies are again making progress in improving the durability of the finishes, thereby improving the durability of the car parts.

Railroad Paints:

The railroads of the United States with their miles of right of way, their thousands of structures, and over two million pieces of rolling stock represent one of the largest outlets for paint products. The conditions to which the various equipment used by the railroad is exposed includes everything from a mild rural environment to one of acid, alkali, water immersion, extreme heat, and abrasion.

The equipment that must be protected includes buildings, signaling devices, rails, bridges, piers, electrification towers, locomotives, passenger and freight cars. The largest individual item of all these in so far as paintable area is concerned is freight cars, and, therefore, this discussion will be concentrated on the types of paint and the methods used to paint these cars. The painting of many of the other items has been covered elsewhere in this paper.

There are about two million freight cars in use by the railroads of the United States. The marketing of paints for freight cars is highly competitive. This competition, coupled with certain reciprocity demands, has relegated quality to a secondary position in most instances. This is most unfortunate since normally most freight car paint systems must give protection for approximately ten years before repainting.

In general, the variety of conditions to which freight cars are exposed has not been fully appreciated, and too often one paint system will be applied to a large number of cars regardless of their intended service. Refrigerator cars, for instance, will be subjected to the corrosive action of brine so that the paint system applied should be formulated to resist this corrosive agent. Coal cars, tank cars, etc., all present specific protective coating problems.

Usually the paints for freight car painting have been inexpensive in cost, the pigment consisting essentially of a cheap grade of iron oxide. The practice of using such paints may be a holdover from the days when freight cars were built primarily of wood. However, the advance to all-steel cars has not brought forth a corresponding demand for high-quality metal protective paints which are essential to really effective protection against corrosion.

The importance of surface preparation of the metal prior to painting has to be given careful consideration in this field of metal protection. The methods used for railroad cars consist usually of sand or shot blasting, thus permitting the use of primers or paints of the faster drying synthetic resin types, since such paints perform well over completely cleaned surfaces.

The undercarriages of all freight cars are of iron and steel construction and are exposed to general atmospheric conditions, excluding sunlight, but have the additional problem of abrasion from flying particles of the roadbed. The undercarriages of most freight cars are painted with one or two coats of an anticorrosive paint plus a topcoat which should be hard enough to withstand abrasion.

The sides and roof are exposed to abrasive action from cinders plus the usual atmospheric conditions of rain, sun, etc. Here the use of poor-quality paints has been quite widespread. However, engineers responsible for maintenance of railroad equipment are recognizing that the exposure conditions call for an anticorrosive paint, such as the red lead pigmented types, and that a final saving will result in the purchase of quality paints.

The interior finishes of freight cars will depend wholly upon the material to be carried. Such materials as coal, ore, cement, oil, solvent, fertilizers, lime soda, chlorine, and acids require paints designed to withstand these particular corrosive actions.

More and more, railroad engineers are subjecting metal protective paints to laboratory and field evaluation tests prior to their purchase. Obviously this procedure will result in benefits to both the railroads and the paint industry.

METHODS OF TESTING

No one in the paint industry, in fact in most industries, doubts the desirability of developing accelerated tests which will duplicate or simulate the results of different types of natural exposure or for a specific end-use.

A great deal of work has been done during the past twenty years by scientific societies and members of the paint industry to develop accelerated test methods for protective organic coatings, but it is still necessary that the final evaluation of paints be made under conditions of actual use. However, it is possible to screen out poor paint systems by the present accelerated test methods.

Modern industrial conditions requiring protective coatings vary widely and are highly special in many cases so that general test methods are often unsatisfactory.

METAL PAINTING

An arbitrary classification of these conditions for metal protective paints would be:

1. Construction, such as building framework and railroad equipment, where conditions may or may not be severe. The atmosphere of certain areas may be more severe than other areas.
2. Industrial
    (*a*) General, such as automobiles, refrigerators, washing machines, and other household appliances not subject to severe conditions.
    (*b*) Plants, such as oil refineries and chemical plants, where corrosive conditions may be very severe.
    (*c*) Lining of tank cars to carry specific liquids such as turpentine, naphtha, hot ammonium nitrate, etc.
3. Marine, including both fresh and salt water shipping, dock structures, locks and dams, where moisture resistance is a greater factor than in (1) or (2).

Although the ferrous metals are the most important from volume use, the various light metals are rapidly increasing in industrial use and will further complicate the problem of adequate protective paint systems and methods of test. The development of a wide variety of synthetic resins has provided the paint technician with new raw materials for protective paints which have marked improvement in water and chemical resistance but are deficient in some physical properties such as methods of application and adhesion. Inhibitive pigments also increase the protective properties by retarding corrosion, but it is difficult to duplicate the exact conditions of actual use in an accelerated test procedure. The absence of a small item may not affect one specific pigment combination but may seriously affect another. Better methods for cleaning and chemically treating metal surfaces are available and new ones are being developed, adding a further complication in the test procedures. The metal industries have contributed a great deal in this respect, but there is room for considerable improvement in the condition of iron and steel fabricated for industrial use.

For metal protection, paints depend for their effectiveness upon three factors: resistance to water or moisture, since water is an essential agent for corrosion; chemical inhibition (not present in all paints or effective under all conditions); resistance to weathering. The relative weight of these characteristics varies with the type of service for which the paint is designed. The accelerated tests measure them either in part, in whole, or in combination. Measurements of single properties are often more valuable than complex tests.

An outline of the accelerated tests now in use will indicate the trend and may stimulate new ideas from this Society, whose fundamental interest is better test methods.

ACCELERATED WEATHERING

Machines are available which provide radiation from electric arcs to simulate sunshine. Panels rotate about an arc and are wet by water spray for rain and changes in temperature for the various seasons. Many added treatments such as ice baths, refrigerated water, immersion overnight in hydrogen peroxide, exposure to oxygen or ozone have been tried. Ice baths and refrigerated water seem to be of some value. Correlation of the rate of failure in this test with outdoor exposure has not been too successful, but much valuable information in regard to rust inhibition, adhesion to metal, and adhesion between coats is gained. McMullen (35) states that the test as described is of some value for paints to be applied on dock structures and on the topside of ships, which are frequently exposed to the effects of water but not totally submerged. This test is of little value for totally submerged exposures for which there seems to be no accelerated test.

HUMIDITY CABINETS

Cabinets may be built to give approximately 100 per cent relative humidity at temperature of 100 F. or higher and have two important variables. The test panels may be simply hung in the chamber or supported on a cold backing which gives large condensation and an accelerated condition. Various corrosive materials may be injected, such as ammonia or sulfur dioxide, to increase further the severity of test. Such cabinets are excellent for batch to batch control. Several days in a cabinet at high humidity and temperature, alternated with 4 to 5 days in the weathering machine, does a fair job of differentiating among paint systems.

SALT FOG SPRAY MACHINES

Salt spray machines are now available in standard forms, but there will always be considerable controversy regarding the kind and amount of salt to use, method of supporting the test panels, etc. Tide water exposure in Florida may not agree with results in the salt spray cabinet or with conditions of actual use, particularly if the ship or metal equipment on the seashore is maintained chiefly in Northern territory. The salt fog spray test will quickly eliminate poor coating systems. It is also of value to determine satisfactory film thickness or coverage necessary for retarding corrosion.

Preliminary observations for marine testing are conducted by submerging coated panels in water in glass tanks. In this way a few paint systems can be chosen from many for testing under actual use condition. It is advisable to pass a current of air through the water to keep up the oxygen content. The water should be the type in which the paint is to be used—approximately 3 per cent salt (preferably sea salt) for ocean water, or natural water of similar mineral content to that in which exposure is to be made. Distilled water is unsatisfactory as it gives results varying widely from natural waters.

It should be remembered that this is in no sense an accelerated test but merely a simulation of natural conditions for easier observation.

CHEMICAL TESTS

Immersion in the corrosive liquid or application of spots of the chemical to the test panel in the laboratory provides a good indication of resistance in actual use. The limitation of this test is the inability to find suitable chemicals to simulate some actual conditions. Human perspiration is one which has not been duplicated and rancid fats are also difficult.

THEORETICAL OR RESEARCH TESTS

The importance of adequate test procedures for materials which protect chemical engineering equipment is being realized to an increasing extent as evidenced by the excellent work being done on the fundamentals involved. Equipment may suffer severely from corrosion fatigue, which in turn accelerates corrosion and together they materially shorten the useful life of the equipment. Measurements of the rate of corrosion under specified conditions and of the ability of coatings to retard this rate are complicated procedures but will undoubtedly provide valuable fundamental information. Another important factor is film continuity. What are the conditions of application or of film thickness which will provide a completely continuous film free from minute pores or imperfections? The work which has been done on this phase of the subject has paid big dividends in satisfactory linings for beer cans, tank cars, and other equipment.

This very brief outline serves to point out that a great deal of very useful work has been done, that accelerated tests will not always indicate results from actual use, and that the field is wide open for new ideas and better test procedures.

It is interesting to mention that about 30 years ago Subcommittee VII on Accelerated Tests for Protective Coatings of A.S.T.M. Committee D-1, had organized for this very important work of metal testing of paint systems. Members represent most of the important commercial enterprise interested in such testing. There is also in the Federation of Paint and Varnish Production Clubs a Standards-and-Methods of Test Committee for the purpose of evaluating methods of tests and standardization. In addition, a joint A.S.T.M.-Federation Committee has been appointed for the purpose of coordinating the efforts of the two societies along this line.

WOOD PAINTING

For exterior paints on wood, some success has been obtained in correlating mainly fading and chalking by accelerated weathering machines as against natural exterior exposure. However, no success has been obtained in producing exactly the same type of failure by the accelerated test methods as in exterior exposures. At the present time accelerated testing of exterior paints merely serves to separate the "sheep from the goats" and to determine trends in variation of composition which are useful in developing superior formulation. Accelerated testing should certainly be followed by actual exterior exposure of coated panels, and then by actually painting houses.

The other tests mentioned for metal painting may be used also with success for special wood finishing systems, which have to withstand humidity, dilute chemical, alcohol, etc.

METHODS OF ANALYSIS

Methods of physical and chemical analysis are finding greater application in the protective coating field. The analytical methods for drying oils have been available for many years for the protective coating industry; resinography and the general analysis of resins are of recent introduction.

"Resinography," as the name implies, is a graphic study of resins. This term is analogous to "metalography" and "mineralography." By revealing morphology of the resinous matrix and of botanical, metallic, and mineral constituents, the method permits correlation of the structure of resins with the physiochemical properties involved in industrial use. The term "resinography" was coined by Theodore G. Rochow and R. L. Gilbert (**44**).

"General Analysis of Resins," Robert W. Stafford and Emil F. Williams (**48**) have made considerable advances as shown in their work, and they point out that the significant steps have been the increasing application of physical methods, as exemplified by the qualitative and quantitative aspects of optical and electron microscopy, ultraviolet (emission and absorption) and infrared spectroscopy, and spectrophotometry. Used together with the purely chemical methods of analysis, these techniques yield otherwise unobtainable information.

PLASTICS *versus* ORGANIC COATINGS

I agree with Blackmore (4), Lurie, Bradley, Fazig (32), and others that, from the viewpoint of chemical composition, both the organic and plastic coatings industries utilize the same raw materials, that is nitrocellulose, ethylcellulose, aceto-butyrate, cellulose acetate, phenol-formaldehyde, urea, melamine, methacrylate, polystyrene, polyethylene, etc. The essential differences between these two industries lie in the methods of formulation and the application of the finished materials.

The plastics technician reduces the raw materials mentioned to a finely powdered state and shapes them in heated molds or melts them and extrudes the molten mass through hot dies. Sometimes he puts them into solution and impregnates paper or fabrics with them. These are subsequently laminated into sheets, rods, or tubes by heat and pressure.

The protective coating technician takes the same materials and puts them first into solution and then applies them to other surfaces by means of brush, dip, roller coat, flow coat, spray gun, or electrodeposition methods. In either case, whether applied by fusion and subsequent freezing or by deposition from a solvent, the end result is exactly the same from a chemical standpoint.

The paint and varnish industries had been making plastic material in their everyday cooking kettle long before the creation of a plastics industry. Aside from all this, it is not hard to see that the technical progress made in one industry will immediately aid the progress of the other. Blackmore (4) states it nicely: "It is unimportant whether the hen came before the egg, as long as there are plenty of both hens and eggs to go around. Similarly, whether or not a product finds its initial use in plastics or paints is unimportant as long as technicians remain alert to its possibilities and potential applications in their respective fields."

STATISTICAL TRENDS

Just how large and how important is the protective organic coating or paint industry? What are the basic chemical trends? How is the industry changing and what about its future? These, and similar questions find an answer in the following statistics.

Table VI gives the total sales in dollar volume of the Paint, Varnish, and Lacquer industry from 1936 to 1945. Although data are representative of 680 establishments reporting, the dollar sales are a fair indication of the total size of the industry. It is quite apparent from these data that the protective coating industry is a well-established basic chemical industry showing a steady and progressive growth. Some prominent and well-informed executives in the paint industry and Mr. Ernest T. Trigg, president of the National Paint, Varnish, and Lacquer Association, are of the opinion that in a few years the annual sales of paint and varnish products will reach a billion dollars.

Table VII gives the consumption of oils and some resins in the paint and varnish industry from 1931 to 1945.

These data cover, as an example only, four of the basic resinous ingredients more commonly used in the manufacture of protective organic coatings. These

TABLE VI.—SALES OF PAINT, VARNISH, LACQUER, AND FILLERS BY 680 ESTABLISHMENTS, 1936-1945.
Source: *U. S. Bureau of the Census*: *Current Statistical Service.*

Year	Percentage of Total Output[a]	Total Sales		Unclassified Sales		Total Classified Sales				
								Industrial Sales		
		Number of Establishments Reporting	Value	Number of Establishments Reporting	Value	Number of Establishments Reporting	Trade Sales	Total	Paint and Varnish	Lacquer
1936	. .	680	$398 032 770	100	$36 738 501	580	$203 073 252	$158 221 017	$111 032 702	$47 188 315
1937	. .	680	419 103 821	100	39 075 074	580	215 868 460	164 160 287	115 295 425	48 864 862
1938	91	680	347 682 114	100	34 673 943	580	195 963 442	117 044 729	81 193 901	35 850 828
1939	91	680	394 508 431	100	36 661 951	580	213 749 249	144 097 231	102 405 167	41 692 064
1940	90	680	412 515 812	100	38 395 402	580	214 155 268	159 965 142	111 718 366	48 246 776
1941	90	680	555 398 819	100	51 829 948	580	276 168 508	227 400 363	162 632 021	64 768 342
1942	90	680	529 745 027	100	57 125 387	580	260 484 650	212 134 990	155 858 623	56 276 367
1943	90	680	568 620 999	100	54 519 594	580	265 201 659	248 899 746	186 186 562	62 713 184
1944	90	680	618 325 532	100	61 147 302	580	294 154 629	263 023 601	197 081 086	65 942 515
1945	90	680	643 425 141	100	66 033 201	580	307 394 294	269 997 646	204 747 066	65 250 580

[a] The establishments whose data are included in this table produced *approximately the above stated percentage* of the total value of the output of the industry as reported at the preceding Census of Manufactures.

TABLE VII.—CONSUMPTION OF OILS AND RESINS IN THE PAINT AND VARNISH INDUSTRY, 1931 TO 1944–1945.

Sources: U. S. Tariff Commission.
Foreign Commerce and Navigation of the United States.
U. S. Bureau of Agricultural Chemistry and Engineering.
U. S. Bureau of the Census.

Year	Production of Alkyd Resins (net), lb.[a]	Imports of Natural Varnish Gums and Resins, lb.	Consumption of Rosin in Paints and Varnishes, lb.	Consumption of Oils in Paints and Varnishes, lb.
1931	. . .	28 077 919		. . .
1932		19 635 234		
1933	9 930 705	30 228 962		297 560 000
1934	15 219 247	24 547 016		329 894 000
1935	34 312 713	36 961 032		394 005 000
1935–36[b]			73 622 500	
1936	46 952 452	37 061 380		429 020 000
1936–37[b]			70 021 500	
1937	58 450 032	45 095 263		458 522 000
1937–38[b]			68 448 500	
1938	37 563 840	28 591 011		358 134 000
1938–39[b]			65 139 000	
1939	70 208 098	42 296 526		423 113 000
1939–40[b]			78 759 000	
1940	91 446 195	54 032 473		434 736 000
1940–41[b]			78 899 000	
1941	139 738 000	138 062 509		570 802 000
1941–42[b]			108 873 000	
1942	103 769 000	65 234 792		511 609 000
1942–43[b]			82 060 000	
1943	205 988 000	23 467 961		444 342 000
1943–44[b]			65 697 000	
1944	239 765 000	46 005 705		536 156 000
1944–45[b]			69 231 000	

[a] Exclusive of all fillers, drying agents, dyes, plasticizers, and solvents. Chemical modifiers such as abietic acid and oils are considered part of the net resin content when they are chemically combined with the principal resin.

[b] Crop year, beginning April 1.

TABLE VIII.—INDUSTRIAL DISTRIBUTION OF SYNTHETIC RESINS, 1941–1944 (NET RESIN IN 1000 LB

(Computed from U. S. Tariff Commission Statistics)

	1941	1942	1943	1944
Total Synthetic Resin Production	452 171—100%	439 999—100%	653 938—100%	784 137—100%
For Use in Protective Coatings	212 077— 47	148 255— 34	249 429— 38	292 566— 37
For Use in Molding and Casting	121 684— 27	122 745— 28	130 416— 20	119 915— 15
For Use in Laminating	46 227— 10	61 058— 14	61 906— 10	56 942— 7
For Miscellaneous Uses[b]	72 183— 16	107 941— 24	222 187— 33	314 714— 41

[a] This information is not available for the prewar years.

[b] For adhesives, for treatment of textiles, paper and leather, for glazing, sheeting, films, ion exchange, etc.

figures illustrate the phenomenal growth of the alkyd resins, the more widely used synthetic surface coating material. However, it is interesting to note that at the same time three other natural constituents have not suffered any losses in volume consumption. Nevertheless, it is pointedly clear from the data that the chemical character of the paint industry is being fundamentally affected by the progress of science. The industry has been alert indeed in adopting the more useful man-made products. It is moving forward and at an ever faster technological pace. The statistical growth of other resinous materials, such as phenolic, amino, and vinyls, could have been used to illustrate the chemical character and the technological trend of the coating industry.

Table VIII illustrates the interesting fact that synthetic resins find not only many and varied types of industrial outlets but also that the paint industry constitutes their largest single consuming market. Over a third of the entire synthetic resin production goes into the preparation of protective coatings. This limited presentation of statistical data is of sufficient indication that the paint industry forms a permanently large and significant segment of the chemical industry. The nature of the industry is such that it is adopting new chemical products and principles without completely discarding the time-tested ones. Old as the paint industry is from the standpoint of art and craftsmanship, it is technologically young, vibrant, and progressive and has evolved into a chemical engineering industry. Its future growth and welfare are closely dependent upon continued and vigorous utilization of the new scientific developments that are appearing on the horizon.

FUTURE TRENDS

Let us briefly peer into the crystal ball and see what the future has in store for the protective coating industry.

Continued and, in most cases, increased consumption of the various synthetic resins now in use is indicated—alkyds, maleics, phenolics, ureas, melamines, vinyls, acrylates, pentaerythritols, polyterpenes, coumarone-indenes, chlorinated rubbers, cellulosic derivatives, etc.

Coatings made from styrene and styrene copolymers, organosilicones, synthetic rubbers, itaconic acid resins, and allyl ester polymers are in the offing. The paint chemists' dream of 100 per cent solids synthetic resin coatings may soon come to fruition through the use of monomeric substances as solvent carriers for alkyds and other polymerizable resins.

At the same time, although most of us seem to agree that the future of the protective coating industry lies in the field of synthetic resins and high polymers, we should not forget that linseed oil is a low polymer raw material. Yet at present it is still the largest volume material used in coatings, particularly in house paints. We should not forget also that while we are all thinking of synthesizing new raw materials, there may be other natural raw materials whose discovery may be worth our while. One such example is Garcia Nutans oil—a hard drying oil with properties superior to China wood oil, quicker drying, and with improved alkali resistance.

Isomerized and chemically processed oils, tall oil, and tall oil derivatives will probably also be utilized in increasingly larger quantities.

The composition of paints, varnishes, and lacquers is not the only phase of the industry which will be affected by the progress of time.

As a result of the constant search for newer and better ways of making paint, present batch manufacturing systems are undergoing modernization. Automatic controls and larger unit processes (in certain cases, even continuous operations) will make for more uniform paint products manufactured at lower costs.

We will also see improved and speedier methods of paint applications. Among them may be cited spraying in an electrically induced field, electrostatic detearing, and high-frequency induction heating.

Emulsion coatings, anti-fungus and insect repellant paints, hot melts, and luminescent finishes came into particular prominence for specific applications during the war period. Many of these uses will carry over into postwar civilian markets. For some of the outlets, paints and plastics will find themselves in direct competition. After all, synthetic ressins are the basic raw materials of both and we are rapidly approaching the time when it will be difficult to differentiate in terminology between paints and plastics, except where the latter are used solely as molding materials.

In short, the paint industry is on the move! It is progressing and changing so rapidly that the one thing we can safely predict is that the coatings of tomorrow will be as different from those of today as the coatings of today differ from those of yesterday.

GENERAL CLOSING REMARKS

Protective organic coatings are a necessary and integral part of all industrial and chemical engineering industries. And because of their wide diversity of uses and applications, the thoughts expressed here are those of the author and also those of his many friends who have helped him immeasurably in the preparation of the lecture.

Although the importance of organic coating for war use has apparently passed, it is noteworthy that probably the most important accomplishment of the protective coating industry during the recent war was its ability to satisfy all the military requirements and, at the same time, take reasonably adequate care of civilian needs. It was a source of gratification to see the close cooperation that existed between the Government agencies and the technical men of the coating industry in all matters, whether preparing specifications for new purposes or revising specifications for old purposes, and particularly so when the aim was to release critical materials such as phthalic anhydride and *p*-phenyl phenolic resin for really urgent needs. Considerable credit is due the production side of the industry for being sufficiently versatile to make the frequent adjustments rendered necessary by the changes in available raw materials and the increase of production required in relatively short periods of time.

The coating industry has advanced chemically much more rapidly in the development of raw materials than in the development of chemical engineering processing of coatings. In the future, more attention will be given to the chemical engineering aspects of the industry.

As already mentioned, there is an increasing trend on the part of the more progressive varnish manufacturers to process in larger units. Many are installing and others are planning to install set kettles of larger capacity (1000 to 5000 gal.) than used before or during the war period. In most cases these kettles will be of the closed type equipped for careful critical control of temperature and the application of vacuum or inert gases. In some instances active gases may be used. This definitely indicates the desire of the protective coating industry to provide better products and to project its efforts beyond the age of craftsmanship into an era where processes and products will be more scientifically controlled.

Our technicians, chemists, chemical engineers, and other technically trained men of the industry are thinking more and more in terms of changing from batch processing to continuous methods of production. They may find it somewhat difficult to produce coatings continuously, in view of the fact that many hundreds of different raw materials are used by a protective coating manufacturer to make the variety of coatings normally required. However, comparatively continuous processing is possible because many of the raw materials used in varnish processing have chemical properties within relatively limited ranges.

The idea of continuous processing will be applicable not only in varnish manufacturing but also in the dispersion of pigments into vehicles for the production of paints, enamels, and other pigmented coatings. Generally speaking, the steps in the manufacture of pigmented coatings are a premixing of the dry pigments into the vehicle and grinding—in reality a further dispersion of the pigments into the vehicle, now usually accomplished by means of a Burr stone mill, roller mill, steel and pebble ball mills, Banbury mill, Bramley mill, or colloid mill. The method of dispersion for pigments in these first two steps—premixing and grinding—should probably make some of the greatest advances in the future. The pigment manufacturers are now giving much more serious consideration to the question of pretreating pigments so that they can be more easily dispersed. A great deal more engineering consideration will have to be given to the mechanical means of dispersing pigments. A wealth of real chemical engineering processing can be accumulated in a study of pigment dispersions in vehicles.

Technical selling, particularly for industrial or specialty product finishes, has become more prominent in the coating industry. Coating manufacturers are now training their industrial sales representatives in the fundamental physical and chemical properties of raw materials, and the effect of these properties in the processing of coatings to meet specific end-use requirements. These men are being thoroughly trained in the various methods of application, that is, brushing, spraying, dipping, flow or roller coating, laminating, etc. A thorough understanding not only of the chemical and engineering aspects of the coating is necessary but also of the engineering requirements of the surface on which the coatings are to be applied. In line with this train of development many coating manufacturers are employing technically trained men for soliciting their industrial and maintenance fields.

There are over 1200 paint manufacturers in the United States. It is an industry of approximately 700 million dollars in sales, which may increase, as already indicated, to a sales volume of a billion dollars. Many of the manufacturers are small. In view of the chemical and engineering nature of the coating industry, however, every manufacturer of paint, varnish, or lacquer, no matter how small, should employ chemists or chemical engineers as a measure of protection for his future technical security, as a part of the industrial growth of our country. The technical members of the industry should join at least one of the national technical societies as an aid to his own industrial and technical growth, and thus be of greater technical usefulness to his employer and his country.

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SYMPOSIUM ON ATMOSPHERIC WEATHERING OF CORROSION-RESISTANT STEELS

ATMOSPHERIC CORROSION TESTS ON HIGH-CHROMIUM STEELS*

By W. O. Binder[1] and C. M. Brown[1]

Synopsis

This paper presents data on atmospheric corrosion tests, based on the weight-loss method, for low-carbon 0 to 18 per cent chromium steels conducted at Kure Beach, N. C., Niagara Falls, N. Y., and New York, N. Y. The test data show a progressive improvement in atmospheric corrosion resistance with increasing chromium content and indicate that an addition of 18 per cent chromium is required to make the steels virtually stainless in the atmospheres tested.

Tension test specimens of cold-rolled austenitic steels of the types used in modern light-weight construction were exposed at Niagara Falls, N. Y., for 5 yr. These steels showed no change in strength or ductility when tested after exposure.

The influence of stress was also investigated in tests made at Amagansett, L. I., N. Y., on cold-rolled austenitic steels, and it was found that the presence of stress did not reduce their resistance to marine atmospheric corrosion.

Steels containing substantial amounts of chromium are finding increased application in the fields where resistance to atmospheric corrosion is important, because of the well-known passivating effect that chromium imparts to steel under oxidizing conditions. Tests conducted by the A.S.T.M. on wire and wire products (**1**)[2] made of 18 per cent chromium, 8 per cent nickel steel and 12 to 14 per cent chromium steel have shown excellent results, with the 18 per cent chromium, 8 per cent nickel steel retaining its metallic appearance, and the 12 to 14 per cent chromium steel showing only a slight rust or rust staining in 5 to 6-yr. exposure to several different urban, marine, and rural atmospheres. The A.S.T.M. is also conducting inspections of architectural installations of corrosion-resistant steel at such locations as New York, N.Y., and Atlantic City, N.J. (**2**). These inspections are showing the 18 per cent chromium, 8 per cent nickel steel to be giving very satisfactory service.

While these results are available in the literature, further data on the effect of high percentages of chromium are important properly to evaluate the effect of this alloying element on the atmospheric corrosion resistance of steel. In this paper it is planned first to present quantitative data on marine atmospheric corrosion tests being conducted at Kure Beach, N. C., and industrial atmospheric corrosion tests at New York, N. Y., and Niagara Falls, N. Y., on a series of steels containing additions of chromium in amounts from 0 to 18 per cent. These data will serve to show the effect of continued additions of chromium in improving the atmospheric corrosion resistance of low-carbon steel. The second portion of this paper will then consider atmospheric corrosion tests on cold-rolled austenitic chromium-nickel and chromium-manganese-nickel steels of the type suitable for light-weight construction.

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[1] Union Carbide and Carbon Research Laboratories, Inc., Niagara Falls, N. Y.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 607.

STEELS WITH 0 TO 18 PER CENT CHROMIUM

The tests on low-carbon steels containing 0 to 18 per cent chromium were begun in 1941 and 1942. In addition to chromium, the steels contained small amounts of manganese and silicon, together with minor amounts of copper, sulfur, and phosphorus. One series of samples was exposed at Kure Beach, N. C., another series at Niagara Falls, N. Y., and a third at New York, N. Y. The steels were made in 30-lb. induction furnaces from commercial materials and were hot-rolled to No. 16-gage sheet. The steels containing up to 2 per cent chromium after hot-rolling were heated 1½ hr. at 650 C. and air-cooled; those containing 3 to 5 per cent chromium were heated 1 hr. at 700 C. and air-cooled; and the remainder were heated 2 hr. at 750 C. and air-cooled. The scale was removed by pickling. Test samples 3 by 6 in. in size were then prepared and carefully weighed. The samples were exposed at an angle of 30 deg. from the horizontal in corrosion-resistant steel racks facing south and were held in place by porcelain insulators. Enough samples were exposed so that separate and complete sets could be removed after definite periods of time up to 15 yr., with no cleaning being done between exposure and final removal of a particular group of samples. Whenever a set of samples was removed from test after its exposure period, visual examination was made of the rust character, after which the samples were electrolytically cleaned in inhibited 5 per cent sulfuric acid solution before final weighing (3). In Table I

TABLE I.—EFFECT OF CHROMIUM ON ATMOSPHERIC CORROSION RESISTANCE OF LOW-CARBON STEELS.

Original Condition: Annealed and Descaled
Panel Size: 3 by 6 by 0.06 in.
Test Locations: Kure Beach, N. C., Niagara Falls, N. Y., New York, N. Y.
Exposure Begun: 1941–42.

Steel	Chemical Composition, per cent							Weight Loss, g. per sq. in.							
								Kure Beach, N. C.			Niagara Falls, N. Y.			New York, N. Y.	
	Carbon	Chromium	Manganese	Silicon	Copper	Phosphorus	Sulfur	6 months	18 months	44 months	6 months	20 months	42 months	18 months	52 months
No. E-922	0.07	....	0.39	0.30	0.070	0.018	0.023	0.15	0.27	0.55	....	0.21	....	....	....
No. E-924	0.06	1.02	0.35	0.23	0.058	0.012	0.015	0.12	0.24	0.46	....	0.17	....	....	....
No. E-929	0.05	2.18	0.56	0.30	0.066	0.012	0.022	0.10	0.19	0.31	....	0.14	....	....	....
No. E-934	0.04	3.15	0.50	0.32	0.072	0.010	0.020	0.10	0.16	0.25	....	0.11	....	....	....
No. E-939	0.07	5.34	0.56	0.32	0.056	0.012	0.022	0.08	0.18	0.19	....	0.11	....	....	....
No. E-943	0.06	7.28	0.61	0.33	0.074	0.016	0.022	0.05	0.10	0.14	....	0.09	....	....	....
No. E-947	0.06	9.32	0.67	0.31	0.076	0.011	0.021	0.02	0.07	0.08	....	0.04	....	....	....
No. E-950	0.06	12.53	0.73	0.31	0.068	0.012	0.018	0.01	0.007	0.01	....	0.02	....	....	....
No. E-951	0.04	18.57	0.70	0.40	0.052	0.012	0.018	0.003	0.001	0.002	....	0.002	....	....	....
No. E-773	0.07	....	0.30	0.10	0.053	0.007	0.025	....	....	....	0.14	0.22	0.31	0.45	0.61
No. E-841	0.05	2.09	0.26	0.15	0.070	0.008	0.026	....	....	....	0.08	0.13	0.17	0.40	0.46
No. E-816	0.03	3.27	0.37	0.20	0.070	0.009	0.023	....	....	....	0.05	0.11	0.13	0.13	0.16
No. E-821	0.07	5.50	0.35	0.17	0.070	0.012	0.022	....	....	....	0.08	0.10	0.12	0.09	0.11
No. E-834	0.06	7.24	0.33	0.20	0.060	0.007	0.023	....	....	....	0.10	0.09	0.10	0.12	0.11
No. E-838	0.06	9.13	0.33	0.18	0.060	0.006	0.023	....	....	....	Nil	0.05	0.06	0.09	0.06
No. 12 Cr	0.08	12.58	0.76	0.46	0.060	0.019	0.014	....	....	....	Nil	0.004	0.009	Nil	0.005
No. 18 Cr	0.08	17.26	0.45	0.32	0.070	0.015	0.014	....	....	....	Nil	0.003	0.003	Nil	0.001

are given the results of these tests to date for the steels in the three test locations.

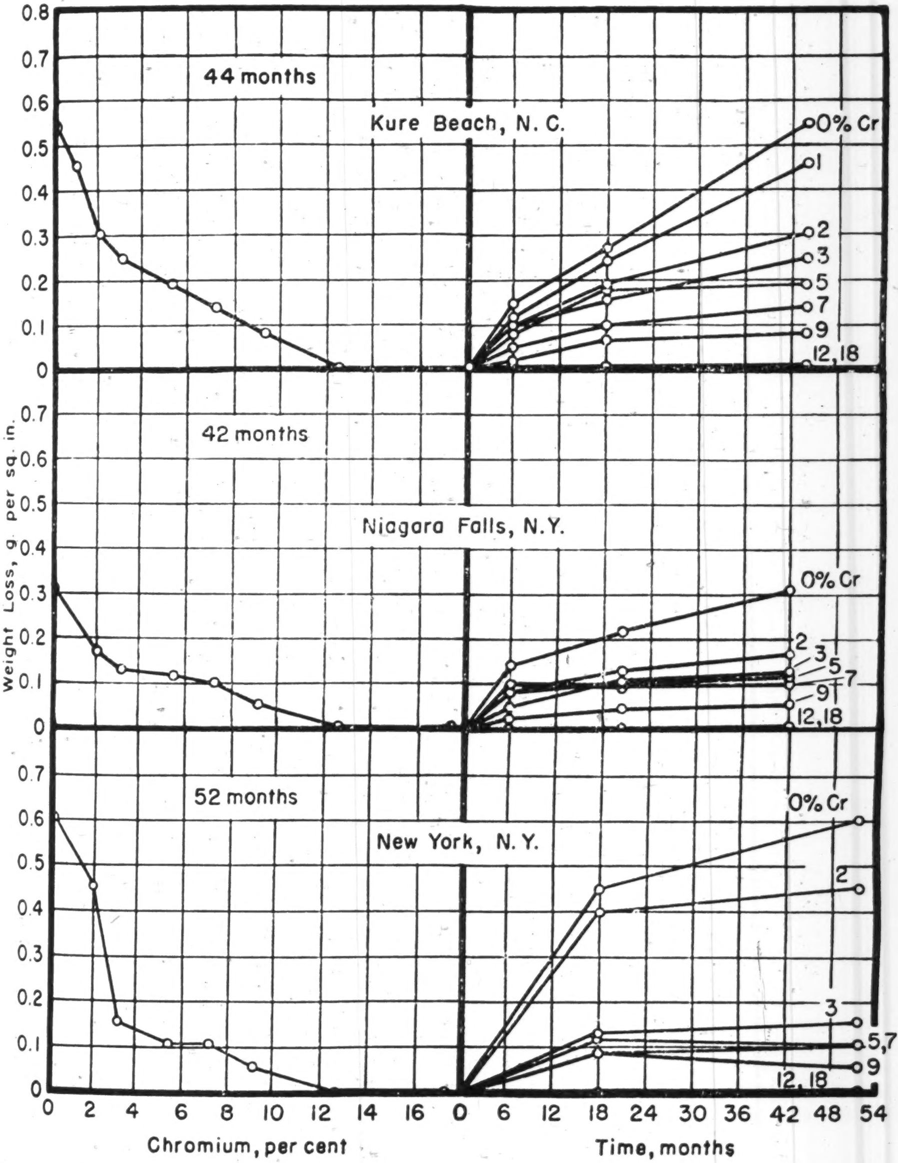
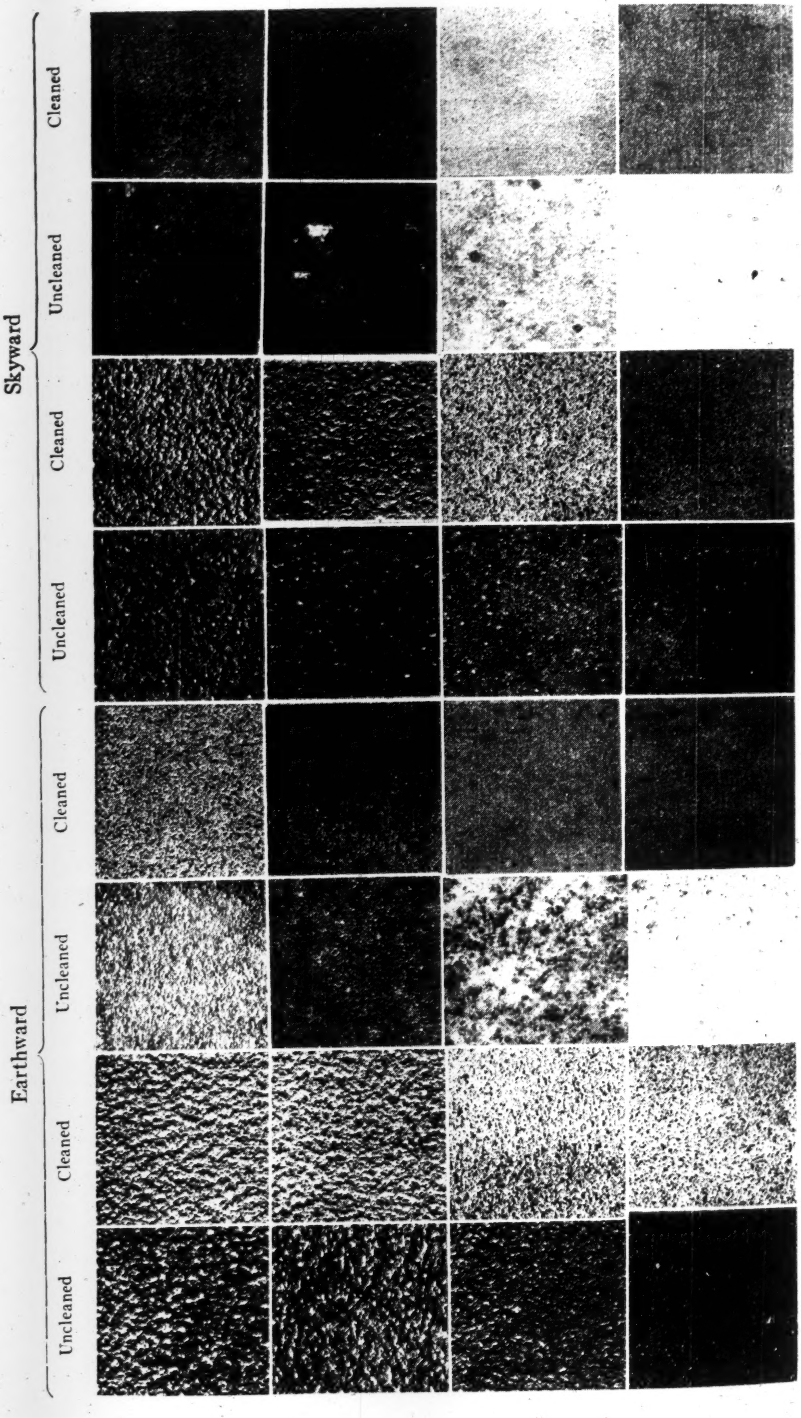


FIG. 1.—Influence of Chromium on the Atmospheric Corrosion Resistance of Low-Carbon Steel.

The test results from Table I are plotted in Fig. 1 as curves of weight loss for the maximum exposure time *versus* percentage of chromium, and as weight loss *versus* time for each chromium content.

In the case of the Kure Beach, N. C., samples, rusting was observed to be quite uniform on both the earthward and


Earthward
Uncleaned
Cleaned
Uncleaned
Cleaned
Skyward
Uncleaned
Cleaned
Uncleaned
Cleaned

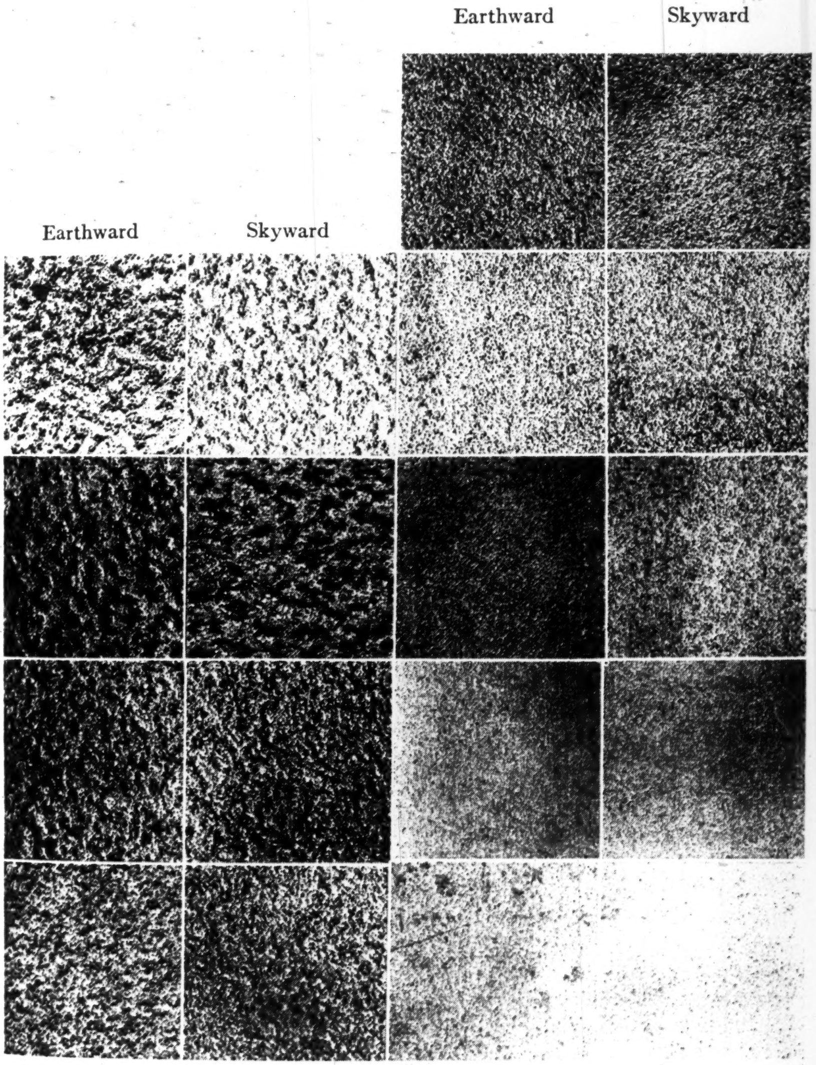
1st row—0 per cent chromium
2nd row—2 per cent chromium
3rd row—3 per cent chromium
4th row—5 per cent chromium

1st row— 7 per cent chromium
2nd row— 9 per cent chromium
3rd row—12 per cent chromium
4th row—18 per cent chromium

1st row—0 per cent chromium
2nd row—2 per cent chromium
3rd row—3 per cent chromium
4th row—5 per cent chromium

1st row— 7 per cent chromium
2nd row— 9 per cent chromium
3rd row—12 per cent chromium
4th row—18 per cent chromium

FIG. 2.—Appearance of Steels After 52 Months' Atmospheric Exposure at New York, N. Y.



1st row—0 per cent chromium
2nd row—1 per cent chromium
3rd row—2 per cent chromium
4th row—3 per cent chromium

1st row— 5 per cent chromium
2nd row— 7 per cent chromium
3rd row— 9 per cent chromium
4th row—12 per cent chromium
5th row—18 per cent chromium

FIG. 3.—Appearance of Steels After 44 Months' Atmospheric Exposure at Kure Beach, N. C., and Cleaning.

skyward faces. The samples exposed at Niagara Falls, N. Y., and New York, N. Y., however, appeared more corroded on the earthward than the skyward side. As the chromium content was increased, the character of the rust changed from a loose, heavy scale to an adherent and a much lighter scale at all locations. At 12 per cent chromium the rusting was very slight, and at 18 per cent chromium it was practically nil and could be removed by washing with soap and water and a mild household cleanser. The 12 per cent chromium steel was very slightly pitted and the 18 per cent chromium steel was practically free from pitting.

Examination of the data for the steels listed in Table I shows that increasing the chromium content of the low-carbon steels decreases the weight loss due to atmospheric corrosion at all three test sites. The weight loss *versus* chromium content curves shown in Fig. 1 indicate a significant change in slope at about 3 per cent chromium and again at about 12 per cent chromium for all locations. In general, the Niagara Falls, N. Y., atmosphere appears somewhat less corrosive than either the Kure Beach, N. C., or New York, N. Y., atmospheres. The New York, N. Y., atmosphere is the most corrosive for the steels containing up to 3 per cent chromium, whereas the Kure Beach, N. C., atmosphere is the most corrosive for the steels containing 3 to 9 per cent chromium. The 12 per cent chromium steel and, in particular, the 18 per cent chromium steel, show a high degree of resistance to rusting at all locations, even though exposed for approximately four years without cleaning. The variable effect of different atmospheres is probably linked with the apparent necessity of having at least 12 and preferably 18 per cent chromium present in steel before universal atmospheric immunity is realized. Steels with lower percentages of chromium are thereby more susceptible to varying results, depending upon the particular local conditions, because their tendency to become passive is not strong enough.

The photographs of Fig. 2 show the condition of the samples exposed for 52 months at New York, N. Y. and Fig. 3 the samples exposed 44 months at Kure Beach, N. C. These photographs are full-size and represent the center section of both skyward and earthward faces as they appeared before and after electrolytic cleaning, except in the case of the Kure Beach, N. C., samples which were only photographed in the cleaned condition. In the New York, N. Y., samples, a general roughening of the surface was noted on the plain low-carbon steel. The surface of the 2 per cent chromium steel was roughened to a lesser extent. At 3 per cent chromium, a marked difference in the character of the clean surface existed, the surface being considerably smoother than the surfaces of the former steels. Beyond 3 per cent chromium, the steels retained more and more of their original surface appearance. A shallow but rather discrete pitting type of attack, previously obscured by the general attack, was observed to begin at about 3 per cent chromium. As the chromium content increased beyond 3 per cent, the size of the pits became smaller, until at 12 per cent they were barely visible to the unaided eye. As previously stated, practically no pitting was observed in the 18 per cent chromium steel. General and decreasing surface roughness was observed in the Kure Beach, N. C., samples as the chromium content approached 5 per cent. The pitting transition observed at New York, N. Y., in the steels containing between 3 to 12 per cent chromium was similarly found in the Kure Beach samples containing 5 to 12 per cent chromium. The 18 per cent chromium steel at Kure Beach showed no pitting after 44 months exposure.

TABLE II.—EFFECT OF 5 YEARS' ATMOSPHERIC EXPOSURE ON TENSILE PROPERTIES OF COLD-ROLLED AUSTENITIC CORROSION-RESISTANT STEELS.
17-7, 18-8, 18-8 Columbium, 18-8 Molybdenum Steels
Test Location: Niagara Falls, N. Y. Exposure Begun: 1941

Steel	Chemical Composition, per cent								Tensile Properties[a]									
	Chromium	Nickel	Manganese	Silicon	Carbon	Nitrogen	Molybdenum	Columbium	Original				Stored 5 yr.			Exposed 5 yr.		
									Condition[b]	Yield Strength, 0.2 per cent Offset, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Yield Strength, 0.2 per cent Offset, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Yield Strength, 0.2 per cent Offset, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent
No. E-380	17.25	7.18	1.21	0.37	0.10	0.05	....	....	2-C	140 000	189 600	20.0	147 000	195 500	19.0	147 000	196 700	19.0
									2-C[1]	161 000	186 200	22.0	165 000	189 100	20.0	155 000	182 800	24.5
									2-D	163 000	196 900	15.0	175 000	207 000	15.5	166 000	201 000	16.5
									2-D[1]	184 000	201 000	15.5	184 000	198 900	14.0	186 000	199 200	14.0
No. D-246	17.43	7.75	0.54	0.35	0.11	0.06	....	....	1-B	122 000	172 300	29.0	110 000	169 200	31.0	110 000	169 000	28.0
									1-C	135 000	186 800	24.0	....	....	....	135 000	186 800	23.0
									1-D	177 000	211 000	11.0	191 000	211 000	15.0	175 000	206 100	14.0
									2-B	97 000	181 400	31.0	113 000	174 500	28.5	103 000	170 000	28.0
									2-C	135 000	189 500	17.5	160 000	198 500	17.0	140 000	191 800	14.5
									2-D	179 000	207 600	13.5	189 000	222 200	10.0	187 000	217 000	16.0
No. E-391	17.25	7.43	1.21	0.41	0.14	0.13	....	....	2-C	130 000	159 000	25.0	133 000	158 000	23.5	134 000	157 500	22.0
									2-C[1]	137 000	156 700	23.0	147 000	165 000	22.0	141 000	161 000	23.0
									2-D	149 000	176 600	9.0	166 000	187 000	9.5	160 000	181 100	12.0
									2-D[1]	169 000	186 500	7.0	179 000	198 000	9.0	172 000	189 200	10.0
No. E-582	18.35	8.27	1.30	0.40	0.06	0.05	....	....	2-C	121 000	157 000	20.0	117 000	152 000	20.0	121 000	154 200	19.0
									2-C[1]	139 000	158 600	15.0	....	....	....	134 000	154 000	19.5
									2-D	136 000	161 200	16.0	139 000	165 100	15.0	141 000	164 100	16.0
									2-D[1]	154 000	171 200	6.0	149 000	172 400	6.0	150 000	173 300	7.0
No. E-381	18.54	8.00	1.24	0.35	0.10	0.03	....	....	2-C	119 000	157 200	19.5	120 000	157 100	20.0	120 000	160 200	20.0
									2-C[1]	136 000	155 800	18.5	133 000	155 500	18.5	132 000	158 500	23.0
									2-D	147 000	174 100	12.0	141 000	167 500	9.0	146 000	171 500	13.0
									2-D[1]	157 000	179 300	6.0	166 000	186 500	5.0	157 000	178 300	7.0
No. E-382	19.72	8.75	1.28	0.41	0.10	0.03	....	....	2-C	118 000	139 400	20.0	118 000	136 600	20.0	114 000	135 800	20.5
									2-C[1]	122 000	138 200	19.5	127 000	144 500	15.0	124 000	141 200	20.5
									2-D	139 000	160 000	9.0	134 000	159 200	7.0	140 000	164 000	8.5
									2-D[1]	147 000	166 200	7.0	145 000	165 500	7.0	143 000	164 600	7.0
No. E-593	18.54	8.47	1.28	0.53	0.07	0.04	....	0.82	2-C	....	....	....	136 000	154 700	9.5	137 000	158 000	4.5
									2-C[1]	....	....	....	145 000	160 000	2.5	128 000	166 000	5.0
									2-D	....	....	....	145 000	162 000	5.0	144 000	165 500	5.0
									2-D[1]	....	....	....	159 000	181 000	3.0	155 000	176 000	2.5
No. D-797	18.05	9.52	0.59	0.52	0.07	0.07	....	0.82	2-C	109 000	140 300	16.0	115 000	140 000	18.5	113 000	145 200	17.0
									2-C[1]	127 000	148 000	10.0	128 000	152 000	8.5	122 000	150 200	11.0
No. E-389	19.00	13.10	2.12	0.49	0.07	0.05	2.12	....	2-C	118 000	133 500	14.0	119 000	133 200	16.0	115 000	130 200	14.5
									2-C[1]	125 000	136 000	12.0	123 000	144 500	15.0	123 000	134 500	11.0
									2-D	129 000	147 000	9.0	148 000	163 000	10.0	138 000	152 600	10.0
									2-D[1]	140 000	152 300	9.0	142 000	155 800	7.0	146 000	156 800	10.0
No. E-390	18.97	13.42	2.05	0.45	0.07	0.05	3.08	....	2-C	118 000	134 000	14.0	124 000	137 000	15.5	123 000	136 200	16.0
									2-C[1]	124 000	134 400	14.0	130 000	141 900	12.5	127 000	137 400	14.0
									2-D	134 000	153 600	9.5	139 000	154 800	10.0	140 000	157 900	8.0
									2-D[1]	148 000	159 400	8.0	150 000	163 900	8.5	149 000	157 000	9.5

[a] Samples taken parallel to rolling direction.
[b] 1—0.06-in. thick strip. 2—0.03-in. thick strip. B—Cold-Rolled 20 per cent. C—Cold-rolled 30 per cent. D—Cold-rolled 40 per cent.
[1] Cold-rolled and heated 72 hr. at 200 C. and air-cooled

TABLE III.—EFFECT OF 5 YEARS' ATMOSPHERIC EXPOSURE ON TENSILE PROPERTIES OF COLD-ROLLED AUSTENITIC CORROSION-RESISTANT STEELS.
Chromium-Manganese-Nickel Type
Test Location: Niagara Falls, N. Y. Exposure Begun: 1941

Steel	Chemical Composition, per cent						Condition[b]	Tensile Properties[a]								
								Original			Stored 5 yr.			Exposed 5 yr.		
	Chromium	Nickel	Manganese	Silicon	Carbon	Nitrogen		Yield Strength, 0.2 per cent Off-set, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Yield Strength, 0.2 per cent Off-set, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Yield Strength, 0.2 per cent Off-set, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent
No. D-853	18.22	4.07	5.00	0.39	0.12	0.07	2-B	....	....	....	115 000	197 000	26.0	113 000	188 200	31.5
							2-B[1]	130 000	183 200	32.0	120 000	184 100	34.0	120 000	182 000	32.5
							2-C	148 000	214 200	19.0	158 000	217 000	18.0	160 000	216 000	16.5
							2-C[1]	169 000	200 200	15.0	173 000	207 900	20.0	171 000	202 000	19.5
No. D-75	18.29	4.12	5.25	0.40	0.10	0.06	1-B	101 000	171 400	27.5	110 000	178 900	32.0	109 000	176 800	33.5
							1-C	142 000	190 000	20.0	160 000	193 200	18.5	158 000	197 800	20.5
							1-D	183 000	201 500	7.0	169 000	199 200	11.0	179 000	203 000	15.0
							2-B	89 000	165 000	25.0	102 000	177 800	25.5	104 000	174 000	27.5
							2-C	133 000	187 800	16.0	145 000	194 800	18.0	139 000	189 100	17.0
							2-D	173 000	203 300	14.0	177 000	212 000	14.0	165 000	201 000	14.5
No. D-787	18.01	4.08	4.84	0.38	0.11	0.12	2-B	97 000	171 400	36.0	107 000	176 500	31.0	101 000	174 800	36.0
							2-C	137 000	203 200	24.0	130 000	196 000	21.0	125 000	191 900	21.5
							2-C[1]	160 000	191 200	19.0	148 000	192 000	26.0	161 000	194 800	19.5
No. D-78	18.07	4.70	4.83	0.39	0.10	0.12	1-B	95 000	150 500	30.0	106 000	156 000	33.0	103 000	155 000	34.0
							1-C	124 000	176 900	20.0	134 000	183 000	22.0	139 000	188 200	21.5
							1-D	156 000	189 800	13.5	162 000	193 800	16.5	168 000	198 000	10.5
							2-B	107 000	168 400	25.5	111 000	168 000	21.0	114 000	167 700	26.0
							2-C	124 000	184 500	18.5	135 000	186 000	20.0	128 000	178 400	21.0
							2-D	147 000	190 300	15.0	151 000	194 200	16.5	149 000	192 900	18.0
No. D-79	18.02	5.19	5.81	0.39	0.10	0.12	1-B	111 000	150 500	31.0	106 000	139 200	32.0	107 000	138 000	29.5
							1-C	....	....	....	138 000	165 100	18.5	133 000	163 200	21.0
							1-D	154 000	183 100	10.0	154 000	183 000	11.0	141 000	182 800	10.5
							2-B	96 000	145 000	27.0	106 000	143 100	25.0	110 000	146 400	28.0
							2-C	119 000	167 600	17.5	134 000	166 000	19.0	125 000	161 800	18.0
							2-D	148 000	177 400	13.0	147 000	176 500	15.0	148 000	179 200	16.0
No. D-791	16.89	6.09	3.78	0.44	0.13	0.13	2-B	105 000	146 600	30.0	119 000	161 000	26.0	118 000	158 800	24.0
							2-B[1]	118 000	152 200	28.0	124 000	154 100	25.0	121 000	150 000	27.0
							2-C	137 000	183 700	18.5	149 000	187 800	18.0	143 000	188 800	19.0
							2-C[1]	160 000	183 600	14.0	160 000	188 000	17.0	157 000	181 200	19.5

[a] Samples taken parallel to rolling direction.

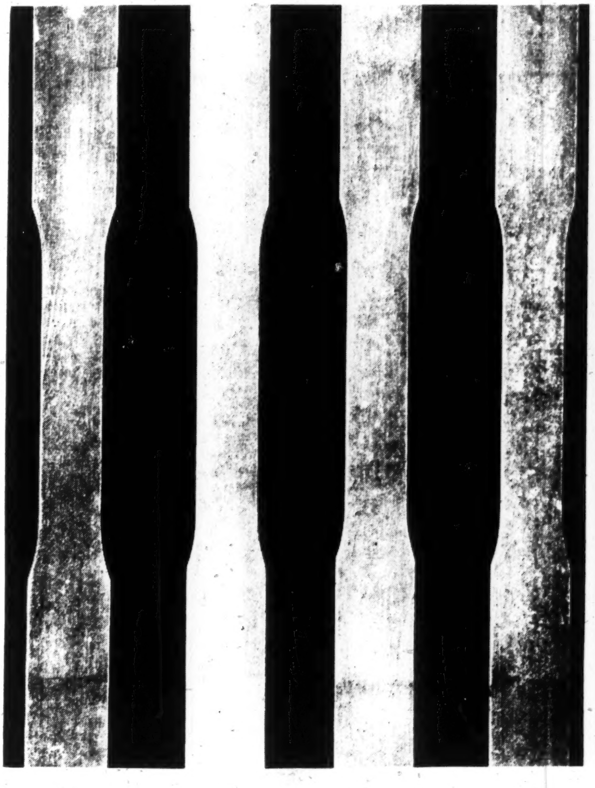
[b] 1—0.06-in. thick strip. 2—0.03-in. thick strip. B—Cold-rolled 20 per cent. C—Cold-rolled 30 per cent. D—Cold-rolled 40 per cent.

[1] Cold-rolled and heated 72 hr. at 200 C. and air-cooled.

AUSTENITIC CHROMIUM-NICKEL AND CHROMIUM-MANGANESE-NICKEL STEELS

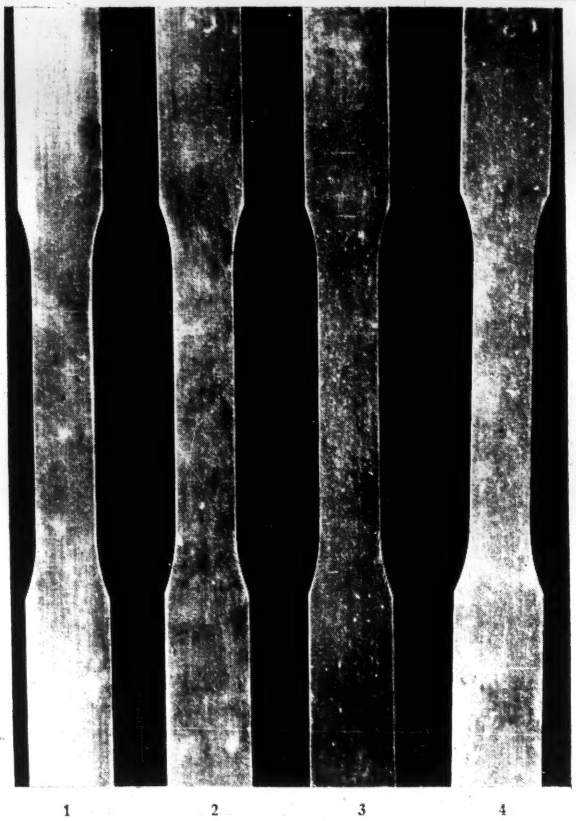
In the tests just described, the 18 per cent chromium steel has been shown to possess practical immunity to atmospheric attack. This steel, however, is not so adaptable for modern light-weight structures as the austenitic 18 per cent chromium, 8 per cent nickel type of steel. Past experience has shown that these steels have been extensively applied in many types of light-weight construction where workability, strength, and corrosion resistance are required, but confirmatory, quantitative data have not been generally available to support this position. For this reason, tests were undertaken about five years ago to expose typical austenitic corrosion-resistant steels in the cold-rolled condition to the atmosphere at Niagara Falls, N. Y.

Because high-chromium steels have been shown to exhibit extremely low weight loss after exposure to the atmosphere, a different procedure for evaluating atmospheric effect was adopted for the austenitic corrosion-resistant steels which were investigated. The method chosen for evaluating the effect of atmospheric corrosion on the austenitic



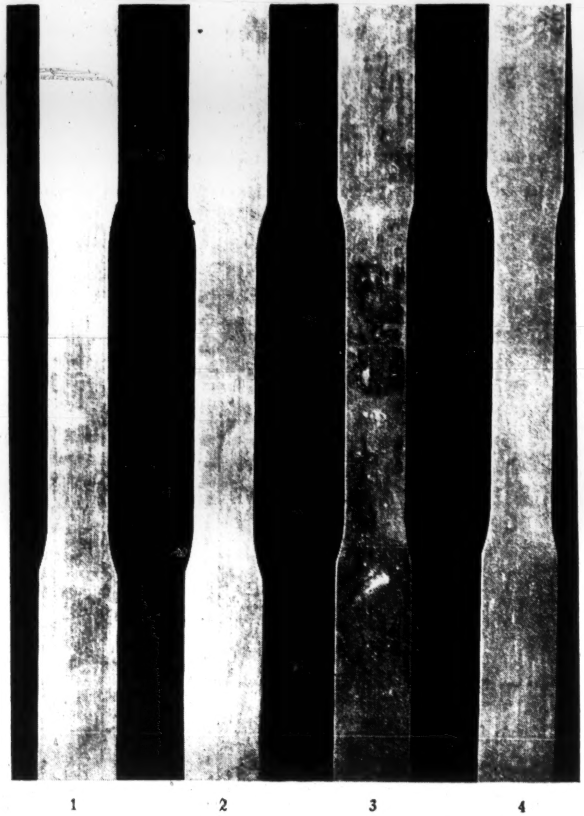
1. Reduced 30 per cent by cold-rolling.
2. Reduced 30 per cent by cold-rolling, heated 72 hr. at 200 C. and air-cooled.
3. Reduced 40 per cent by cold-rolling.
4. Reduced 40 per cent by cold-rolling, heated 72 hr. at 200 C. and air-cooled.

FIG. 4.—Appearance of Earthward Side of Steel E-380 Containing 17.25 per cent Chromium, 7.18 per cent Nickel, and 0.10 per cent Carbon After 5-yr. Atmospheric Exposure at Niagara Falls, N. Y., and Cleaning.



1. Reduced 30 per cent by cold-rolling.
2. Reduced 30 per cent by cold-rolling, heated 72 hr. at 200 C. and air-cooled.
3. Reduced 40 per cent by cold-rolling.
4. Reduced 40 per cent by cold-rolling, heated 72 hr. at 200 C. and air-cooled.

FIG. 5.—Appearance of Earthward Side of Steel E-381 Containing 18.54 per cent Chromium, 8.00 per cent Nickel, and 0.10 per cent Carbon After 5 yr. Atmospheric Exposure at Niagara Falls, N. Y., and Cleaning.



1. Reduced 20 per cent by cold-rolling.
2. Reduced 20 per cent by cold-rolling, heated 72 hr. at 200 C. and air-cooled.
3. Reduced 30 per cent by cold-rolling.
4. Reduced 30 per cent by cold-rolling, heated 72 hr. at 200 C. and air-cooled.

FIG. 6.—Appearance of Earthward Side of Steel D-853 Containing 18.22 per cent Chromium, 4.07 per cent Nickel, 5.00 per cent Manganese, and 0.12 per cent Carbon After 5 yr. Atmospheric Exposure at Niagara Falls, N. Y., and Cleaning.

steels was the tension test method. Several grades of cold-rolled austenitic chromium-nickel and chromium-manganese-nickel steels were included in the tests. Accordingly, in January, 1941, tension test specimens having an 0.5 by 2.25-in. reduced section machined from 0.03 and 0.06-in. thick strip were exposed at Niagara Falls, N. Y., in wooden test racks facing south at an angle of 30 deg. from the horizontal. It was believed that exposing the actual tension specimens would constitute a more severe test than exposing sheet panels from which to machine tension specimens after conclusion of the test. Exposure of actual tension specimens would allow edge corrosion to take place, as well as corrosion of the surfaces, and thereby more greatly affect the tensile properties. Tension tests were originally made on the various steels prior to exposure of the test specimens. To evaluate unknown effects of aging of the steel, a third set of samples was stored in an unheated building a short distance from the test racks. At the end of 5 yr. the exposed samples and the stored samples were tested in tension with the results shown in Tables II and III.

When removed from test, these samples were covered with an appreciable quantity of dirt which was removed with soap, water, and a mild cleanser before photographing and testing. The steels shown in Figs. 4, 5, and 6 are representative 17 per cent chromium – 7 per cent nickel, 18 per cent chromium – 8 per cent nickel, and 18 per cent chromium – 5 per cent manganese – 4 per cent nickel steels as they appeared when cleaned. After cleaning, the steels were examined closely for pitting. In some cases, pitting of a mild nature was observed that was not severe enough appreciably to mar the appearance of the cold-rolled surface. The samples heat-treated 72 hr. at 200 C. and air-cooled after cold rolling behaved similarly to the corresponding unheat-treated samples.

It is observed from the tension test data that 5-yr. exposure to the industrial atmosphere of Niagara Falls, N. Y., has not affected the strength or ductility of these steels, as the differences noted are believed to be within the experimental error for the test. The expected error of measurement and testing of the specimens is probably in the vicinity of ± 4000 to 5000 psi. in the tensile strength determination, and hence the test results cannot be evaluated more closely than dictated by these limits. In determining the percentage elongation, the error is probably within ±0.5 per cent. There is, however, another difficulty in that fractures which occur outside the middle third of the reduced section lead to low elongation figures. Inasmuch as only single specimens were exposed, check tests are impossible in situations of this type.

TABLE IV.—EFFECT OF ATMOSPHERIC EXPOSURE ON TENSILE PROPERTIES OF SPOT-WELDED COLD-ROLLED COMMERCIAL 18 PER CENT CHROMIUM-8 PER CENT NICKEL STEEL,

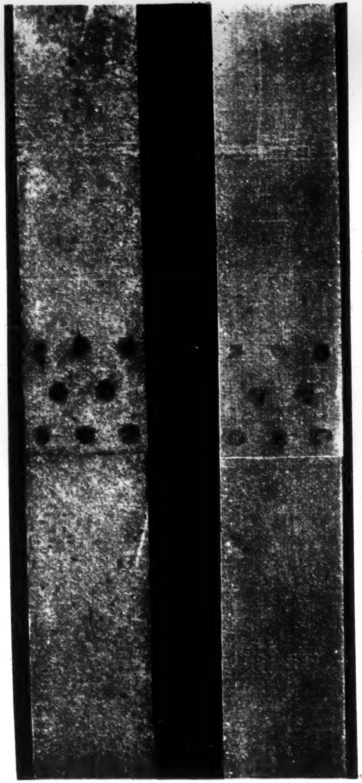
Test Location: Niagara Falls, N. Y.

Exposure Begun: 1935

Type of Sample	Condition of Steel	Sheet Thickness, in.	Length of Exposure, months	Beam Load, lb.
Strips joined by 8 spot welds..	Cold-rolled	0.010	..	1960[a]
Strips joined by 8 spot welds..	Cold-rolled	0.010	12	1920[a]
Corrugated strips joined by 15 spot welds........	Cold-rolled	0.015	..	6800[a]
Corrugated strips joined by 15 spot welds.......	Cold-rolled	0.015	12	6500[a]

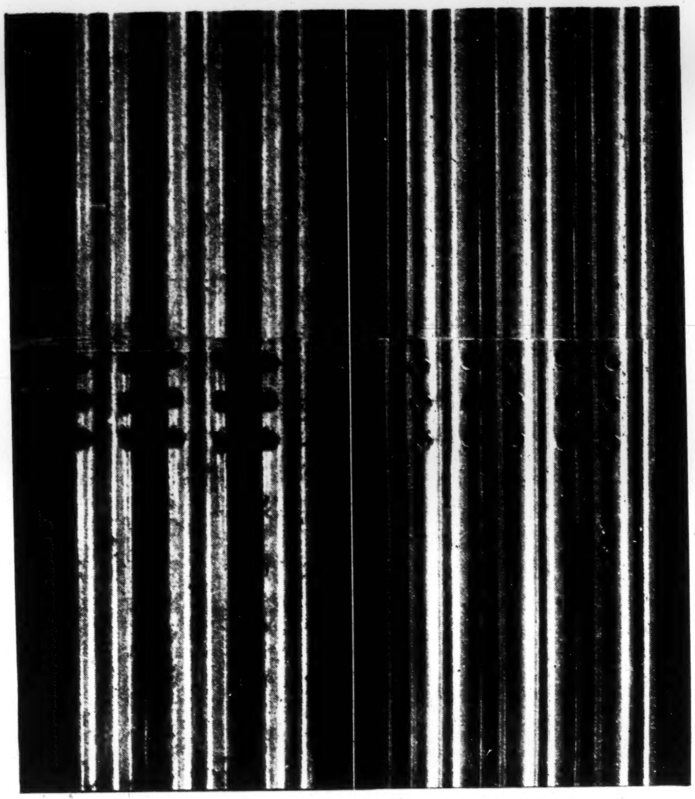
[a] Maximum load in pounds.

Further evidence that the austenitic corrosion-resistant steels are very resistant to atmospheric attack was also obtained from 12-month exposure tests



Before cleaning. After cleaning.

FIG. 7.—Appearance of 0.01-in. Thick 18 per cent Chromium - 8 per cent Nickel Steel Strip Joined by Eight Spot Welds After 12 Months' Atmospheric Exposure at Niagara Falls, N. Y.



Before cleaning After cleaning

FIG. 8.—Appearance ~~of Cold-Rolled~~ 0.015-in. Thick Corrugated 18 per cent Chromium - 8 per cent Nickel Steel Strip Joined by Fifteen Spot Welds After 12 Months' Atmospheric Exposure at Niagara Falls, N. Y.

conducted at Niagara Falls, N. Y., on commercially fabricated spot-welded and corrugated sections. The test results and photographs of the exposed samples before and after cleaning with soap and water are given in Table IV and Figs. 7 and 8. These tests indicate that the steels were not permanently stained as a result of exposure and that their strength properties were practically unimpaired.

EFFECT OF STRESS

Another type of test was also carried out on the beach at Amagansett, L. I., N. Y., to evaluate the effect of stress on the corrosion resistance of the austenitic steels when exposed to marine atmosphere. At this location a rack was set up at a 30-deg. slope from the horizontal facing the ocean and stressed specimens of 0.03 to 0.04-in. thick cold-rolled austenitic corrosion-resistant steels installed. The rack was set approximately 800 ft. from the water. Table V contains the data on the composition and mechanical properties of these steels. The samples were stressed in several different ways to include specimens stressed in both the elastic and plastic ranges. Specimens with sheared edges, punched holes, Erichsen impressions of 3-mm. to 6-mm. depths, 90 and 180-deg. bends around a radius equal to three times the thickness of the sheet were used as representative of plastic stresses. Stressed horseshoe-shaped samples were used as representative of elastic stresses applied to previously plastic-stressed metal, as occurs when formed parts are service-loaded. The strips for the horseshoe-shaped samples were machined to $\frac{5}{8}$ by 3 in. and were formed into circular-shaped arcs whose distance across the free ends was 2 in. Prior to forming, small holes were drilled on the center line $\frac{1}{8}$ in. from each end of the strip. After forming, a 25 per cent chromium-20 per cent nickel steel wire was passed through the holes and external stress was applied by tightening the wire until the curvature of the horseshoe conformed with that of $1\frac{3}{4}$-in. diameter bar. The final type of specimen was prepared as a constant strength cantilever beam stressed by dead loading to calculated values of 30,000, 60,000, 80,000, and 110,000 psi. The dimensions of this specimen are shown in Fig. 9. The deflection produced by the actual loading was maintained during test by a screw arrangement. Before exposure, all specimens were passivated in 20 per cent nitric acid at 65 C. for 30 min.

TABLE V.—MECHANICAL PROPERTIES OF AUSTENITIC CORROSION-RESISTANT STEELS EXPOSED AT AMAGANSETT, L. I., N. Y.[a]

Exposure Begun: 1943

Steel	Chemical Composition, per cent					Condition	Yield Strength 0.2 per cent Offset, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent
	Chromium	Nickel	Manganese	Silicon	Carbon				
No. H-491	16.96	7.07	1.12	0.33	0.12	[b]	154 000	189 300	19.5
No. H-194	17.52	4.34	5.43	0.27	0.12	[b]	164 000	213 000	19.0

[a] Parallel to cold-rolling direction.

[b] Cold-rolled 35 per cent to 0.035-in. thickness.

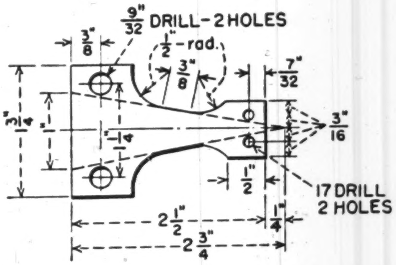


FIG. 9.—Description of Cantilever Type Specimen for 0.03 to 0.04-in. Thick Sheet.

After somewhat more than two years in test, the samples were removed for inspection. A slight rust film was observed on the specimens at the edges, probably indicating the course of drainage. This film was easily removed with soap and water or a mild cleanser. Figures 10, 11, and 12 show the samples after cleaning in this manner. Examination of the samples revealed that none of them had been cracked or otherwise affected by the combination of stress and atmospheric corrosion, as compared with unstressed samples and unstressed portions of the same samples. Some tiny pits were observed in the samples, but these were scattered and apparently had no relation with the way in which the steels were stressed. This pitting attack was somewhat greater in the austenitic chromium-manganese-nickel steel than in the austenitic chromium-nickel steel,

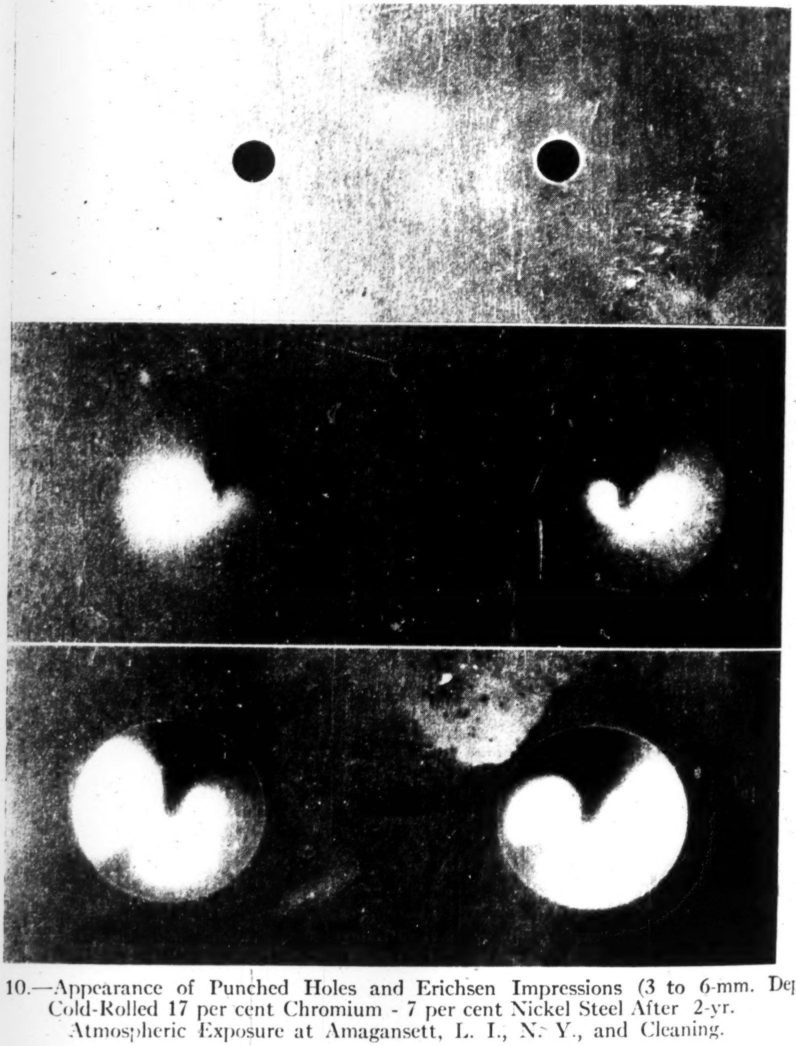


FIG. 10.—Appearance of Punched Holes and Erichsen Impressions (3 to 6-mm. Depth) in Cold-Rolled 17 per cent Chromium - 7 per cent Nickel Steel After 2-yr. Atmospheric Exposure at Amagansett, L. I., N. Y., and Cleaning.

but in neither case was the appearance of the steels appreciably impaired.

SUMMARY AND CONCLUSIONS

This investigation includes data on atmospheric corrosion tests made at Kure Beach, N. C., Niagara Falls, N. Y., and New York, N. Y., showing the increase in atmospheric corrosion resistance obtained by additions of chromium in low-carbon steel. These data bring out that the rate of improvement in atmospheric corrosion resistance changes at about 3 per cent chromium and again at about 12 per cent. An addition of about 18 per cent chromium is required to render the steels virtually stainless in the atmospheres tested. In agreement with other investigations, a difference in corrodibility of the three atmospheres is observed for steels with less than 12 per cent chromium. Tests made at Niagara Falls, N. Y., on cold-rolled austenitic chromium-nickel and chromium-manganese-nickel steels of the type used in light-weight construction indicate no loss in strength or ductility as the result of 5-yr. exposure to this industrial atmosphere. Marine atmosphere tests at Amagansett, L. I., N. Y., on stressed samples of cold-rolled austenitic corrosion-resistant steels indicate that stress does not influence the excellent atmospheric corrosion resistance of these steels.

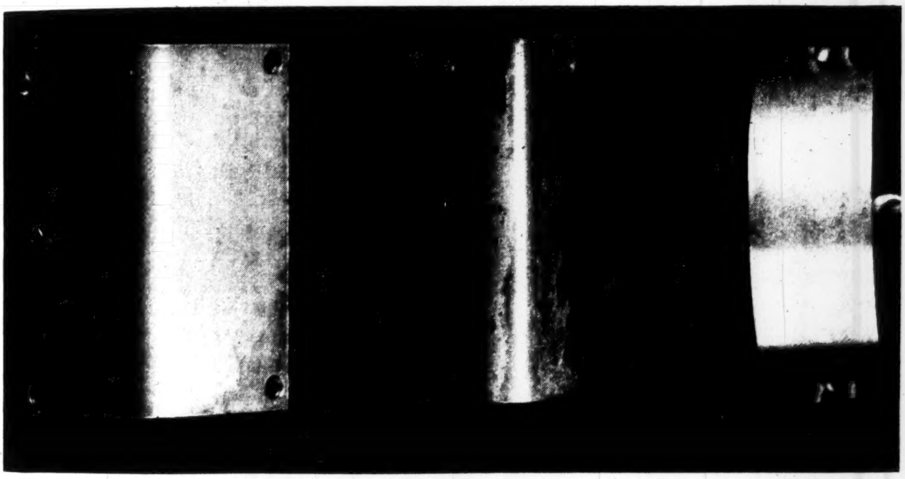


FIG. 11.—Appearance of 90 and 180-deg. Bend Samples and Stressed Horseshoe-Shaped Samples of 17 per cent Chromium - 7 per cent Nickel Steel After 2-yr. Atmospheric Exposure at Amagansett, L. I., N. Y., and Cleaning.

Acknowledgment:

The authors wish to acknowledge the assistance of Russell Franks, C. R. Bishop, J. Thompson, and L. C. De'Aeth of the Union Carbide and Carbon Research Laboratories, Inc., for their aid in carrying out these tests.

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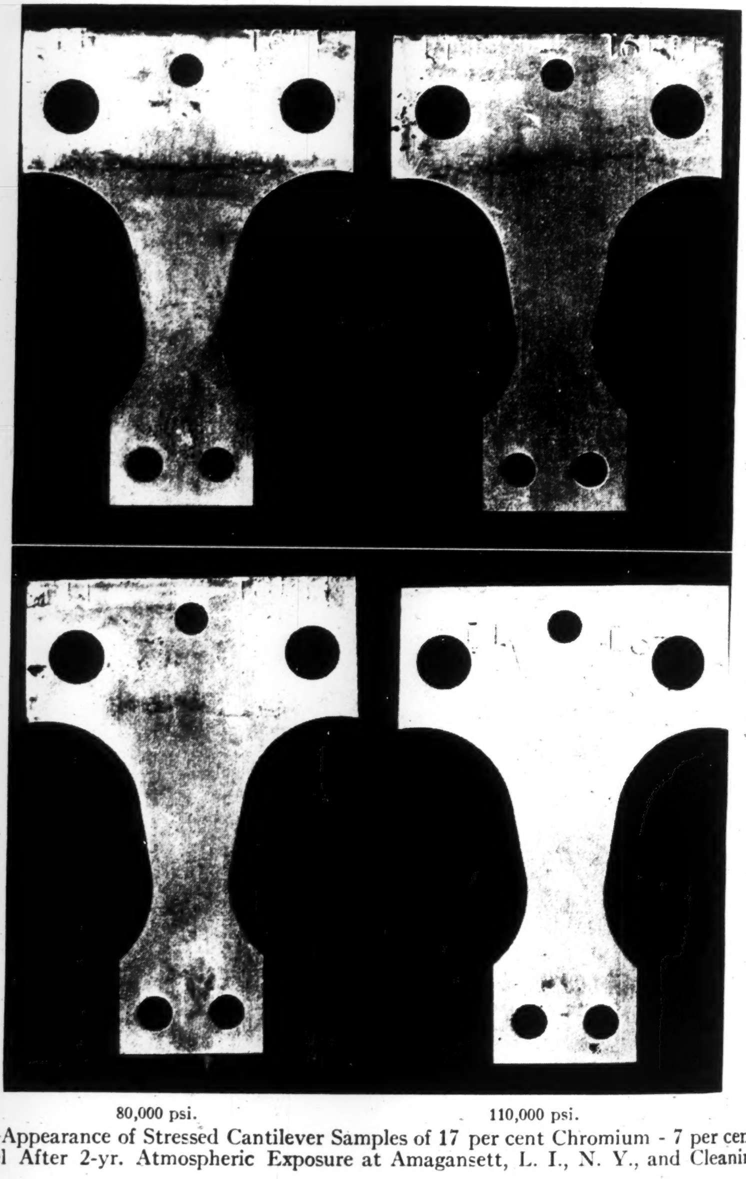
(2) Report of Special Subcommittee on Inspection of Stainless Steel Architectural Structures, *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 197 (1939).

(3) "Corrosion, Processes—Factors—Testing," The International Nickel Co., New York, N. Y., p. 24 (1944).



30,000 psi. 60,000 psi.

80,000 psi. 110,000 psi.

FIG. 12.—Appearance of Stressed Cantilever Samples of 17 per cent Chromium - 7 per cent Nickel Steel After 2-yr. Atmospheric Exposure at Amagansett, L. I., N. Y., and Cleaning.

DISCUSSION

Mr. F. N. Speller.[1]—The data presented on 3 per cent chromium steel remind me of some unpublished results of laboratory and field tests received recently in a private communication from a well-known French investigator. The specimens he had under study contained about 1 per cent aluminum with about 2 and 3 per cent of chromium. Comparison was made with soft open-hearth steel and copper-bearing steel, but he apparently did not include chromium steels without aluminum. However, the losses he reported in atmospheric tests and in half immersed and fully immersed tests in fresh and salt water were considerably less on these chromium-aluminum steels under all conditions of exposure.

The results reported by Mr. Binder and from the Kure Beach tests are particularly interesting to me as they confirm data obtained from a series of laboratory tests in 1924 with steels of various chromium contents which indicated that $2\frac{1}{2}$ to 3 per cent chromium in low-carbon steels gave a distinct break in the curve.[2] This low alloy steel is unique in that it shows considerably less corrosion than ordinary soft steel or copper-bearing steel under *both* atmospheric and underwater exposures.

Messrs. W. O. Binder and C. M. Brown (*authors' closure by letter*).—It is of interest to learn from Mr. Speller of the results of certain French investigations which compare favorably with those given in this paper. Unfortunately no 2 to 3 per cent chromium steels with aluminum were included in this work for direct comparison with the data mentioned. It is also gratifying to know that the corrosion *versus* chromium content relationship as set forth here is in substantial agreement with Mr. Speller's tests made twenty-odd years ago.

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[1] Metallurgical Consultant, Pittsburgh, Pa.

[2] F. N. Speller, "Corrosion Causes and Prevention," McGraw-Hill Book Co., New York, N. Y. (1935). See p. 135.

CORROSION-RESISTANT STEEL FOR ARCHITECTURAL AND STRUCTURAL APPLICATIONS*

By H. A. Grove[1]

In order to obtain information on the behavior of corrosion-resisting steels, under conditions of actual service, a special committee was formed in 1937 to inspect several architectural installations. The special committee visited and inspected the installations at the Chrysler and Empire State Buildings in New York, N. Y., The Philadelphia Saving Fund Society Building in Philadelphia, Pa., and several building fronts along the boardwalk at Atlantic City, N. J. These were reinspected in 1944 by the committee, which also examined a Budd corrosion-resistant steel train which had been in service for several years.

Discussion of Results of Inspections

Chrysler Building, New York, N. Y.:

During the examination of the corrosion-resistant steel on the Chrysler Building, the committee was accompanied on both occasions by the Superintendent of Maintenance for the building.

The steel on the spire, the committee was told, has not been cleaned since 1930 and, as far as could be ascertained, it still appears to be in very good condition. The gargoyles have been cleaned three times since 1930 and they also are in good condition. The stainless-type trim around the windows of the store fronts on the ground level is washed every eleven days at the time the windows are cleaned. All the corrosion-resistant steel on these store fronts is cleaned twice a year with a mixture of Italian pumice and whiting to remove traffic film, while the main entries to the building are cleaned four times a year. All this trim is in a good condition.

Locations on two sides of the building were examined from the seventy-first floor. Inspection revealed that although the steel was covered with a dark film due to the accumulation of smoke and soot, this film could easily be removed with the use of a mild abrasive, showing the original bright finish beneath. All joints and seams had been soldered, and after soldering, the flux had been neutralized with soda. It was interesting to note that there was no corrosion as a result of stray soldering fluxes.

The superintendent stated that the corrosive conditions are very severe at the Chrysler Building, due to the prevailing southeast wind which blows the soot and smoke continuously toward the building from the nearby East River power plants.

The corrosion-resistant steel material on the Chrysler Building is 18 per cent chromium-8 per cent nickel with a No. 4 finish, corresponding to steel grade 2, type 302, finish No. 4 as set forth in A.S.T.M. Standard Specifications for Corrosion-Resisting Chromium-Nickel Steel Plate, Sheet, and Strip (A 167 – 44).[2]

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] Central Alloy District, Republic Steel Corp., Massillon, Ohio.

[2] 1944 Book of A.S.T.M. Standards, Part I, p. 479

An analysis of dirty coating scraped off the Chrysler Building above the gargoyles was made by the Metropolitan Refining Co., Inc., which submitted the following on the results of the tests: carbon (oily soot), 52.1 per cent; sulfates, 29.7 per cent; silica, 10.1 per cent; and the remaining 8 per cent was mainly organic matter and undetermined matter, containing also some chlorides and some iron.

Empire State Building, New York, N. Y.:

The corrosion-resistant steel spandrels on the Empire State Building are of grade 1 type 302 steel with a No. 6 finish (Tampico brushed)[2] and extend from the sixth floor to the observation tower. The building superintendent, with whom the committee consulted, stated that the stainless-type steel had not been cleaned or polished since it had been placed in service in 1931, except for the original cleaning after the building was constructed. At that time the building was washed down with pumice and ammonia water to remove building materials such as plaster, cinders, cement, etc. This cleaning operation was performed before completion of the building and was a regular step in the procedure.

The corrosion-resistant steel mullions and vertical strips were examined at the sixth floor offset and at the seventy-second floor offset. There was no rusting or staining, but there was a dark film present such as that noticed at the Chrysler Building. Removal of this film disclosed that the original finish of this metal was not impaired.

After the bomber crash in 1945, sections taken from the vertical mullions and examined by several laboratories showed the same dirty film that had been noticed at the sixth and seventy-second floors. When this film was removed with cleanser, the surface was found to be in as good a condition as when the building was erected.

An analysis of the atmosphere of New York, N. Y., made by the Bell Telephone Laboratories at the time of the 1937 inspection was as follows: carbon dioxide, approximately 0.04 per cent; sulfur dioxide, 0.8 to 2 ppm.; and chlorides, trace.

Results[3] of exposure of a large number of non-ferrous metals and alloys under the supervision of Subcommittee VI on Atmospheric Corrosion, of A.S.T.M. Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys, have established that the atmosphere of New York, N. Y., is one of the most corrosive in the United States. Evidently the usual corrosion by an industrial atmosphere is aggravated by the chlorides present.

Philadelphia Saving Fund Society Building:

In the Philadelphia Saving Fund Building, steel of grade 1 type 302,[2] with Nos. 4 and 6 finishes, has been used as store fronts, marquees, panels in entry ways, elevator panels, escalator trim, facing of bank vaults, safety deposit rooms, facing for safety deposit boxes, and kitchen equipment in the bank kitchen.

The building was opened in May, 1932. The manager of the building stated at the time of the last inspection (1944) that he was quite pleased with the appearance of the corrosion-resistant steel and the small amount of maintenance work that is required to keep it in first class condition. The cleaning time for outside trim averages approximately one man hour per day. An expenditure of about $12.00 per year covers cost of cleaner and rags for keeping the entry way in condition. The corrosion-resistant steel on the exterior of the building around the windows is washed down at the same time as the windows, which keeps it in good condition. The steel used as interior trim, such as panels, doors, etc., is in excellent condition and shows no sign of tarnish or corrosion.

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[3] Report of Subcommittee VI on Atmospheric Corrosion Tests of Non-Ferrous Metals and Alloys, *Proceedings*, Am. Soc. Testing Mats., Vol. 44, p. 224 (1944).

Atlantic City Buildings:

Corrosion-resistant steel has been used here on building fronts as window moldings, marquees, signs, doors, entry-way panels, and trim. Atmospheric conditions are quite severe due to the close proximity to the ocean and the salt spray which is ever present.

The following store fronts along the boardwalk were inspected: Du Pont Exhibit Building, M. Gettleman, Apollo Theater and Regis Restaurant.

On all store fronts where the stainless type steel is cleaned regularly, the metal is in fairly good condition. At a height above which the window washer cannot reach, and where the metal receives no cleaning, small pits and some rusting can be observed. The depth of the pits was of no consequence and there were no signs of perforation of the metal.

On a store front of M. Gettleman, installed in 1933, both grade 4 type 430 (17 per cent chromium) and grade No. 1 type 302 (18 per cent chromium-8 per cent nickel) were included. The 17 per cent chromium steel discolored quickly and has been replaced by 18 per cent chromium-8 per cent nickel type. This store was recently examined again and found to be badly discolored by rust. This is chiefly on under surfaces where proper drainage cannot occur, thus allowing the atmosphere to dry on it. The worst rusting has occurred since 1945, when there was a fire in the adjoining building. The manager of M. Gettleman plans to have all the corrosion-resistant steel cleaned thoroughly this year.

It was the opinion of the committee that conditions at Atlantic City were more conducive to corrosion and tarnishing of metals than at either New York or Philadelphia because of the salt spray which is constantly blowing on the building fronts. Evaporation permits the salt to precipitate, and, if this precipitate is not removed within a reasonable time, it results in appreciable staining and some shallow pitting. The 18-8, type 302, is more suited for applications of this nature than the 17 per cent chromium, type 430. Comparative tests of a variety of compositions have shown that the chromium-nickel-molybdenum alloy, type 316, is even better suited for use in marine atmospheres. Best performance of any composition will be favored by regular cleaning and by details of construction that provide proper drainage.

"Stainless" Steel Train "Crusader:"

The streamline train Crusader was built by the Edward G. Budd Manufacturing Co., Philadelphia, Pa., and operates on the Reading Line between Philadelphia and New York. It was inspected in the Philadelphia yards, where a thorough examination of the exterior and interior revealed no sign of corrosion. The under carriage of the train is not cleaned and consequently, at the time of inspection, was covered with dirt, grease, and tar from the road beds. Some of this coating of dirt was removed and the corrosion-resistant steel beneath it was found to be in perfect condition.

In general, these trains are of spot-welded construction, and upon examination, these welds showed no staining or corrosion in the welds themselves, or in areas adjacent to the welds. The interior of the train is in excellent condition, although it has received no more than the regular cleaning that ordinary trains receive. It would appear from the examination of this train that corrosion-resistant steel is well suited for this line of transportation.

Corrosion-Resistant Steel Deck Houses:

Two destroyers, the Alwin, commissioned in 1934, and the Farragut, commissioned in 1935, were fitted with stainless-type steel deck houses made by the spot-welded process of the Edward G. Budd Manufacturing Co. This was a Navy experiment to save weight by the use of very light gages of corrosion-resistant steel. These destroyers have been in continuous service for approximately ten or eleven years and during a great part of the war cruised between the Aleutians and Australia, where they weathered several typhoons. At present they have been called home for decommissioning and scrapping within the next few months.

Although the deck houses were constructed primarily of corrosion-resistant steel, they contained some sections made of both carbon steel and aluminum. All of these materials were coated with paint for camouflaging. The corrosion-resistant steel portions of the structures were found to be in especially good condition, showing no signs of corrosion.

Several locations throughout the destroyers were fitted with "stainless" steel, such as door locks, steps, galley equipment, etc. In every instance, this steel gave a good account of itself.

SUGGESTED PRACTICE FOR DECORATIVE INSTALLATIONS

In reviewing the effects of atmospheric corrosion on store fronts, the following recommendations are suggested.

In the development of a satisfactory corrosion-resistant steel store front, care and judgment should be exercised in selecting the proper type of material, surface finish, design, and application. Surfaces should be vertical, wherever possible, to permit easy draining, thus avoiding stagnant pools. All areas should be readily accessible to facilitate cleaning.

As a result of many tests and much experimental work, store fronts and molding sections are produced from sheets or strip, or both. If sheets are used, either a No. 4 or a No. 7 finish is generally used, which is satisfactory since the sheets are ground and polished at the mill. If this surface is not disturbed during fabrication, it is quite suitable for service. Particular care should be taken in processing. Forming dies should be covered with paper or cloth to protect the polished surface against scratching or iron contamination. If strip is used in a rolled molding, as is usually the case, the section must be refinished by brushing and buffing or polishing and buffing to produce a surface clean and bright for outside exposure. Generally speaking, a good brushing with a Tampico brush and grit stick, followed by buffing to a high color and luster, will produce a very satisfactory finish. Buffing alone should not be depended upon.

In some cases, cleaning in nitric acid before polishing is desirable, especially in perforated sections where the brush or buff will not reach all surfaces.

In designing a store front, drainage and cleaning are very important. If the surfaces are mainly vertical, whether plain or fancy, they will be exposed to both rain and sunshine and will, therefore, be subject to natural washing, draining, and drying. This will do some cleaning and is highly desirable, especially in well-elevated areas where frequent cleaning is impossible. Recessed areas will not benefit by such washing, but will tend to collect dirt.

DISCUSSION

Mr. C. H. Sample[1] (*presented in written form*).—Answers to the following questions would be of interest:

1. Was there any evidence of galvanic action between the aluminum and the steels?
2. What was the paint adhesion to each of the three materials used for the deck housing?
3. What were the general and relative appearances of each of the three materials in the deck houses?

Mr. Russell Franks.[2]—I should like to have some comments on the possibility that galvanic effects may have influenced the behavior of the three materials combined in the deck houses with respect to both general performance and the occurrence of any stress corrosion.

Mr. R. B. Gunia.[3]—As one of the inspectors of the deck houses in question, I can corroborate the statements made by Mr. Grove. There was evidence of blistering and lack of adherence of paint to all three materials. In the absence of this protection, certain sections of the aluminum sheathing and structural members had suffered much more severe damage than might be inferred from Mr. Grove's restrained reference to the "oxidation" of the aluminum.

Chairman F. L. LaQue.[4]—The following comments are offered with regard to the questions raised with respect to galvanic corrosion.

Some investigations have shown that "stainless" steels in galvanic couples are less damaging to aluminum than other corrosion-resistant alloys might be. Quantitative data bearing on this point will be made available in the results of tests being completed currently by Subcommittee VIII of A.S.T.M. Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys.

It may be noted also that observations on magnesium cast around a steel core and exposed to a marine atmosphere and sea water spray at Kure Beach, N. C., have shown that the galvanic effect does not extend more than $\frac{3}{16}$ in. from the line of contact. Consequently, in a structure such as the deck houses under discussion, it would be unlikely that one of the metals would exert a galvanic effect on the others that would be observable more than a fraction of an inch from the contact.

Mr. J. T. MacKenzie.[5]—The paper just given by Mr. Mutchler of the National Bureau of Standards showed very severe corrosion, both of aluminum and magnesium when coupled with "stainless" steel in the atmosphere. If aluminum can be used to protect ordinary steel in the ground, it should have an even greater effect on "stainless" steel.

Mr. Carl Buck.[6]—Mr. Grove spoke of the corrosion of "stainless" steel exterior surfaces of buildings exposed to the salt air atmospheres of the shore line where these surfaces were inaccessible for cleaning by window washers. He stated further that when these apparently affected surfaces were cleaned, there was no evidence of pitting or other attack. It occurs to me that the discoloration of the "stainless" steel may have been no more than the result of iron contamination from super structures rather than the result of corrosion of the "stainless" steel. I believe that it is important that this point be established.

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[1] Chief Engineer, Rheem Research Products, Inc., Baltimore, Md.

[2] Technical Service and Development Dept., Electro Metallurgical Co., Pittsburgh, Pa.

[3] Manager, Stainless Steel Bureau, Carnegie-Illinois Steel Corp., Chicago, Ill.

[4] In Charge of Corrosion Engineering Section, Development and Research Division, The International Nickel Co., Inc., New York, N. Y.

[5] Chief Metallurgist, The American Cast Iron Pipe Co., Birmingham, Ala.

[6] Metallurgical Engineer, Stainless Steel, Carnegie-Illinois Corp., Pittsburgh, Pa.

MR. H. A. GROVE (*author*).—So far as could be determined, there was no galvanic action (either acceleration or retardation of corrosion) between the metals in the deck houses. Where galvanized fastenings had been used in the "stainless" steel structures (contrary to recommended practice) there was some discoloration of the "stainless" steel by rust bleeding from the galvanized parts. In general, the distribution of different metals throughout the structure was such that significant galvanic effects would not have been anticipated.

While the paint adhered to the galvanneal better than to the aluminum or the "stainless" steel, there was blistering of paint on all three metals. The greater roughness of the galvanneal appeared to be the principal factor in the better adhesion of paint to it.

The galvanneal showed some rusting, while that portion of the aluminum which had not been replaced during the life of the deck houses showed a certain amount of corrosion which might be called "aluminum rusting" or "oxidation."

There was no evidence of stress corrosion of either the aluminum or the "stainless" steel.

With regard to Mr. Buck's question with respect to the "stainless" steel on the boardwalk store fronts at Atlantic City, it should be noted that corrosive conditions are exceptionally severe with respect to "stainless" steels or other materials attacked by chlorides. In some cases, as Mr. Buck suspected, the discoloration of the "stainless" steel evidently was due principally to bleeding of rust from ordinary steel structural members located above the "stainless" steel or backing it up.

In other instances, there were opportunities for accumulation of pools of salt water in undrained crevices. Like other materials, including glass, "stainless" steel should be cleaned at regular intervals. Even glass is subject to attack by corrosive atmospheres if left uncleaned. Wherever "stainless" steel was used along the boardwalk where it was not, or could not be, cleaned at regular intervals, it showed some corrosion.

MR. S. B. ASHKINAZY.[7]—I should like to ask the several authors what their reaction is in regard to the need for passivating the straight chromium type of "stainless" steels. There seems to be some difference of opinion as to the need for passivating a straight chromium type of "stainless" steel due to the fact that it is expected to tarnish anyway on exposure, whereas in the austenitic type, one of the functions of passivating is the removal of any foreign matter that may have lodged in the steel which might be the start of tarnishing and corrosion.

MR. I. V. WILLIAMS.[8]—I have pretty definite personal feelings on this subject. I do not believe that passivation on a straight chromium "stainless" steel does any good. In most of my experience it is definitely harmful, particularly if the parts so passivated are to be used in any kind of a bearing surface. My own experience has been that parts which are machined under normal machine shop operations are much better off if they are left as they come from the machine with a slight film of oil on them than they are if they are passivated, both as to the effect on the corrosion resistance and most certainly as to the effect on wear resistance which might occur later. We have had a lot of trouble with that.

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[7] Material and Process Standards Supervisor, Sperry Gyroscope Co., Inc., Great Neck, N. Y.

[8] Bell Telephone Laboratories, Inc., New York, N. Y.

MR. ASHKINAZY.—Our own practice at Sperry is that we passivate only the austenitic type of "stainless" steel and do not passivate the straight chromium type. Every once in a while some engineer will ask why we do not passivate the straight chromium type. It makes it rather difficult when steel companies put out literature showing the type of bath to be used and actually recommending passivation of the straight chromium type of "stainless" steel. I do not recall whether it is Allegheny Ludlum or some other firm that has recently sent out additions to their technical manual recommending that the straight chromium type "stainless" steel be passivated.

MR. FRANKS.—I should like to answer just one question. I think that these remarks should be tempered by the question of chromium content. I will agree that if the steel contains as much as 20 or 25 per cent chromium there may or may not be any necessity for treating it with nitric acid to remove impurities, but I am quite certain that the twelve per cent chromium steel will rust unless the iron contamination is removed.

We have encountered this problem in preparing such articles as files for taking care of finger nails, and other kinds of "stainless" steel instruments. These hardened and tempered "stainless" steel articles have rusted while in storage or when put into stock for sale. So, I think what you say is fine if you will define the amount of chromium you are talking about.

MR. WILLIAMS.—I was talking almost entirely of the type 410 and type 420 "stainless" steel. I do not know why, but my experience has been exactly the opposite to yours. The passivated type 410 "stainless" steel has actually rusted over night. In fact, in one case where we had to present it to the Navy Inspector, it was good in the evening when we passivated it and the next morning when we took it to show it to him, it was rusty.

MR. FRANKS.—How much nitric acid did you use to passivate it?

MR. WILLIAMS.—I do not know. It was passivated in the shop.

MR. FRANKS.—That is where the trouble is, because the type of passivating treatment is important. If you use a relatively mild passivating agent just to remove oil or dirt without disturbing the polished or machined surface, you will find the results are very much different.

MR. BUCK.—The word passivating, in my opinion, has been somewhat maligned. A belief which I support is becoming more commonly accepted—that if a "stainless" steel surface is chemically clean, regardless of whether this be accomplished by nitric acid or other appropriate media, exposure to air following cleaning provides optimum corrosion resistance.

MR. GRANT L. SNAIR.[9]—I should agree with Messrs. Franks and Buck on that score. I believe a so-called passivating solution of dilute nitric acid acts merely as a cleaning agent to remove foreign matter from surfaces of steel rather than to impart a passivating effect to the steel. All of our specimens for atmospheric corrosion tests were thoroughly cleaned before exposure. Some of them were passivated by treatment with a 10 per cent nitric acid solution. We found there was no difference in the resistance to corrosion offered by the passivated and nonpassivated specimens.

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[9] Research Chemist, Allegheny Ludlum Steel Corp., Brackenridge, Pa.

MR. H. S. RAWDON.[10]—May I just add a word to what Mr. Franks has said. Some years ago at the National Bureau of Standards we were called in by the Treasury Department having charge of Fort Knox. The walls there, where all the gold of the country is stored, are made of "stainless" steel. They were having trouble with them as to corrosion and did not know what to do. It turned out they had not attempted to clean them at all. A slight acid cleaning was all that was necessary to render them perfectly "stainless."

MR. FRANKS.—I should like to add one thing. In this particular instance which I referred to, we found that a simple low-temperature heat treatment after the article was cleaned was sufficient to develop the continuous film, which Mr. Buck spoke about and which helped the steel materially.

CHAIRMAN LAQUE.—I suspect that in a reasonable amount of time this film will form upon exposure to the atmosphere. To supplement remarks already made by these gentlemen, I should like to remind you that during the past couple of years, at the request of our Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys, we made a survey of current practices and recommendations with respect to the passivation of "stainless" steel. Those have been circulated among the industry at least three or four times. Apparently they are now satisfactory to every one and the summary of the present state of the art of passivation, the reasons for it, and the technique for doing it, will be found in the 1946 report of Committee A-10.[11]

With regard to Mr. Williams' statement and in connection with the functioning of this passivated "stainless" steel, at the time the question originally came up, there was some suspicion at that time that the particular acid treatment used was causing some superficial etching of the straight chromium "stainless" steel. Subsequently it came out that by proper manipulation of the composition of passivating solutions and the supplementary use of chromates or dichromates, that superficial etching could be avoided and perhaps we can be satisfied as Mr. Franks said if the proper acid bath is used.

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[10] Chief, Division of Metallurgy (Retired), National Bureau of Standards, Washington, D. C.

[11] This volume, p. 171.

ATMOSPHERIC CORROSION TESTS OF CORROSION-RESISTANT STEEL WIRES[*]

BY A. P. JAHN[1]

This paper presents the nature and the results of the atmospheric corrosion tests on wire and wire product specimens of corrosion-resistant steel after exposure for about nine years at test sites of various types, including severely industrial, mildly industrial, seacoast, and rural. These tests are part of a larger test on bare steel, zinc-coated steel, lead-coated steel, copper-covered steel, and corrosion-resistant steel wires and wire products contributed by Subcommittee VIII on Field Tests of Metallic Coatings of Committee A-5 on Corrosion of Iron and Steel.

Specimens of unfabricated wire and of farm field fence, made from the following alloys of corrosion-resistant steel are being tested:

Group A.—12 to 14 per cent chromium, 0.12 per cent carbon (maximum), air quenched, tempered to 1100 F., cold drawn to approximately 140,000 psi. tensile strength.

Group B.—12 to 14 per cent chromium, 0.12 per cent carbon (maximum), drawn to finished size, then air quenched to approximately 175,000 psi. tensile strength.

Group C.—18 per cent chromium, 8 per cent nickel, 0.12 per cent carbon (maximum), cold drawn to approximately 300,000 psi. tensile strength.

Specimens of 14-gage chain-link fence of the 18 per cent chromium-8 per cent nickel steel, are also under test.

Table I shows the location and the nature of the specimens involved, and Table II shows the detail data of the specific lots of wire from which the specimens were made.

The effect of corrosion on specimens is being measured by the following:

1. Visual examination at the test locations either semiannually or annually, depending on the corrosiveness of the test site atmosphere. Inspection records are qualitative descriptions prior to the time of rusting and quantitative estimations after the appearance of rust or rust-like corrosion products.

2. Tension tests on the unfabricated wire specimens. These are specimens exposed in groups of seven comparison samples taken from the same lot of wire. An attempt is made to remove and test the first specimens of each heptad when it has lost some 5 to 10 per cent of its original strength.

TABLE I.—OUTLINE OF EXPOSURE TESTS.

Exposure Sites	Number of Specimens Exposed		
	Unfabricated Wires[a]	Farm Fence	Chain-Link Fence
Pittsburgh, Pa.	70	none	1
Sandy Hook, N. J.	70	none	1
Bridgeport, Conn.	70	3	1
State College, Pa.	70	3	1
Ithaca, N. Y.	70	3	1
Lafayette, Ind.	70	3	1
Ames, Iowa	70	3	none
Manhattan, Kans.	70	3	none
College Station, Tex.	70	3	none
Davis, Calif.	63	3	1
Santa Cruz, Calif.	63	3	1

[a] Also referred to as tensile strength specimens. The 70 (or 63) specimens are made up of 7 specimens each of 10 (or 9) wire lots. The seven constituting each lot are alike in composition, preparation, and gage.

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[*] Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

[1] Bell Telephone Laboratories, Murray Hill, N. J.

TABLE II.—UNCOATED CORROSION-RESISTANT STEEL WIRE SPECIMEN LOTS.
Exposed for Atmospheric Corrosion Test (A.S.T.M. Subcommittee VIII of Committee A-5)

Wire Lot[a]	Diameter, in.	Gage	Breaking Load, lb.	Tensile Strength, psi.	Elongation in. 10 in., per cent	Chemical Analysis of Bare Metal				
						C	N	Mn	Cr	Si
No. 001[b]	0.105	12	1230	141 800	3.0	0.08	0.26	0.32	12.50	....
No. 004	0.148	9	2420	140 700	3.5	0.08	0.26	0.32	12.50	....
No. 005	0.053	17	320	140 200	2.1	0.08	0.26	0.32	12.50	....
No. 002[b]	0.098	12½	1310	174 700	4.2	0.07	0.26	0.32	12.50	....
No. 006	0.135	10	2480	173 100	4.0	0.07	0.26	0.32	12.50	....
No. 007	0.047	18	280	163 700	4.7	0.07	0.26	0.32	12.50	....
No. 003[b]	0.073	15	1280	304 800	0.5	0.06	8.73	0.31	18.70	....
No. 008	0.106	12	2510	285 600	2.3	0.06	8.73	0.31	18.70	....
No. 009	0.035	20	290	290 000	...	0.06	8.71	0.31	18.70	..
No. 015	0.081	14	1250	240 400	0.8	0.18	9.10	0.55	18.70	0.40

[a] **Group A.—Wire Lots Nos. 001, 004,** and 005 are 12 to 14 per cent chromium, cold drawn.
Group B.—Wire Lots Nos. 002, 006, and 007 are 12 to 14 per cent chromium, air quenched.
Group C.—Wire Lots Nos. 003, 008, 009, and 015 are 18 per cent chromium-8 per cent nickel.
[b] Also exposed on all wires of farm field fence of the same number.

TABLE III.—REPORTS OF INSPECTIONS OF UNFABRICATED WIRE SPECIMENS.

Abbreviations and symbols used to designate appearance or states of corrosion: M = metallic; G = gray; Y = yellow; R = rust; D = dark (dirt or soot); MG = intermediate between M and G; GY = predominately gray, but showing indication of Y; SY or SR indicates respectively superficial Y or R.

Location[a]	Sample Group[b]	Appearance for Various Approximate Periods of Exposure								
		1 yr.	2 yr.	3 yr.	4 yr.	5 yr.	6 yr.	7 yr.	8 yr.	9 yr.
Pittsburgh, Pa.	A	G	G	GY	GY	GY	D	D	D	D
	B	MG	G	G	GY	G	D	D	D	D
	C	M	M	M	MG	M	D	D	D	D
Sandy Hook, N. J.	A	Y	Y	GY	Y	Y	Y	Y	Y	Y
	B	GY	GY	GY	Y	Y	Y	Y	Y	Y
	C	M	M	M	M	M	M	M	G	G
Bridgeport, Conn.	A	MG	G	G	G	G	GY	G	G	Y
	B	MG	MG	MG	MG	MG	G	G	G	GY
	C	M	M	M	M	M	M	MG	MG	MG
State College, Pa. Lafayette, Ind.	A	M	M	M	M	M	M	M	M	M
	B	M	M	M	M	M	M	M	M	M
	C	M	M	M	M	M	M	M	M	M
Santa Cruz, Calif. Davis, Calif. College Station, Tex.	A	SR	SR	SR	SR	SR	SR	SR	SR	SR
	B	SR	SR	SR	SR	SR	SR	SR	SR	SR
	C	M	M	M	M	M	MG	MG	MG	MG
Manhattan, Kans. Ithaca, N. Y.	A	M	MG	MG	MG	MG	MG	MG	MG	MG
	B	M	MG	MG	MG	MG	MG	MG	MG	MG
	C	M	MG	M	M	M	M	M	M	M
Ames, Iowa	A	SY	SY	SY	SY	SY	SY	SY	SY	SY
	B	MG	MG	M	M	SY	M	SY	SY	SY
	C	M	M	M	M	M	M	M	SY	SY

[a] Where two or more locations are grouped together, all the data refer to each location individually.
[b] The group A, B, or C each includes all the wires of that particular alloy. There were not sufficient differences among wires of the same group to warrant individual reporting.
Group A.—Wire lots Nos. 001, 004, and 005—12 to 14 per cent chromium, cold drawn.
Group B.—Wire lots Nos. 002, 006, and 007—12 to 14 per cent chromium, air quenched.
Group C.—Wire lots Nos. 003, 008, 009, and 015—18 per cent chromium-8 per cent nickel.

UNFABRICATED WIRE

Up to the present time (9 yr. of exposure) no marked corrosion of the wires has been noted. In general, the 18 per cent chromium-8 per cent nickel wires retained their metallic luster. The 12 to 14 per cent chromium types have shown some discoloration or yellowing or rust spotting, but it was superficial in all cases. In Table III are listed the reported results of the annual visual examinations. These results have been summarized from the more detailed data published in the Committee A-5 reports.[2]

The reports of superficial yellowing (SY) or superficial rusting (SR) appearing in Table III, or in other parts of this paper, for the Santa Cruz, Davis, and Ames locations should not be currently interpreted as definite evidence that these locations are corrosive ones or that our corrosion-resistant wires are undergoing significant deterioration. Inspections at these college sites are made by the resident college faculty people—a service much appreciated by Committee A-5. Their descriptions of what they observe are entirely reasonable, but these descriptions may not be identical with those of our A-5 itinerant inspectors who work together in inspecting several sites in the northeast section of the United States.

During the period of the test two specimens of each wire lot were removed for tension tests. These were taken from the Pittsburgh site, the most corrosive test site. One set of wires was taken after 4.9-yr. exposure and the other set was tested after 8.5-yr. exposure. None of the wires from either set showed any significant change in tensile strength or in elongation from its original condition at the start of the exposure test.

FARM FIELD FENCE

Group A cold drawn specimens and group B air-quenched specimens of 12 to 14 per cent chromium appeared about the same, with a slight advantage from an appearance standpoint in favor of the air-quenched specimens. The nine years' inspection records for farm field fence No. 001 (cold drawn) and farm field fence No. 002 (air-quenched) are summarized below:

Bridgeport.—Metallic or metallic gray first year, progressing to grayish yellow at 9-yr. exposure.

State College and Lafayette.—Metallic or metallic gray throughout the test.

Ames, Manhattan, and Ithaca.—Superficial yellowing reported after 3-yr. exposure.

Santa Cruz, College Station, and Davis.—Superficial rusting reported in first year and at all subsequent inspections.

Group C specimens of farm field fence made of 18 per cent chromium-8 per cent nickel (No. 003) were reported metallic or metallic gray at all locations throughout the test, except for superficial yellowing at College Station after 3-yr. exposure.

CHAIN LINK FENCE

The 18 per cent chromium-8 per cent nickel specimen retained its metallic luster throughout the test at all locations, except that at Pittsburgh the specimen was black with soot after 6-yr. exposure.

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[2] Report of Wire Inspection Committee on Field Tests of Wire and Wire Products, Report of Committee A-5, *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 70 (1945); Vol. 43, p. 78 (1943); Vol. 41, p. 101 (1941); Vol. 39, p. 101 (1939); Vol. 37, Part I, p. 117 (1937); Vol. 34, Part I, p. 159 (1934).

CORROSION-RESISTANT STEEL SHEET IN MARINE ATMOSPHERES*

BY WILLARD MUTCHLER [1]

SYNOPSIS

Thin corrosion-resistant steel sheets were exposed to the weather for periods up to three years, in marine atmospheres, at Hampton Roads, Va., Kure Beach, N. C., and Chapman Field, Fla. Information was obtained regarding some effects upon the corrosion of factors such as (1) the presence of small quantities of stabilizing elements in steels containing approximately 18 per cent of chromium and 8 per cent of nickel, (2) locality of exposure, (3) shot-welding, (4) surface finishes and treatments, and (5) contact with light metal alloys. Some data are also contained which reveal the behavior of duplicate panels exposed intermittently or continuously in sea water.

Flexural fatigue tests were used to evaluate the corrosion damage and revealed that the most rapid corrosion occurred during the first 6 months of exposure, and that thereafter the rate of attack was much slower. Most of the alloys investigated behaved similarly and usually varied less than ± 3 per cent in their mean loss in fatigue limits. Steels containing molybdenum as a stabilizing element were much less susceptible to formations of surface rust on weathering than any of the others investigated.

The investigation which forms the basis of this paper was conducted by the National Bureau of Standards under the sponsorship of the National Advisory Committee for Aeronautics, the Army Air Forces of the War Department, and the Bureau of Aeronautics of the Navy Department. These agencies were interested primarily in the corrosion of thin corrosion-resistant steel sheets for use in aircraft likely to be exposed to marine atmospheric conditions, or to be wetted frequently with sea water. The sponsors, with the cooperation of manufacturers, selected the steels for testing, specified the methods used for evaluating the damage from corrosion, and are publishing[2] the results in detail elsewhere.

The principal objective was to establish the relative resistance to corrosion of chromium-nickel alloy steels of the 18-8 type with and without small additions of molybdenum, columbium, and titanium as alloying elements. Information also was desired on the effect of locality of exposure, of shot-welding, of various surface treatments and finishes, and of contact with dissimilar metals.

MATERIALS AND METHODS OF TESTING

The majority of the 0.018-in. thick sheets (Table I) were bright cold-rolled to tensile strengths between 150,000 and 200,000 psi., and were passivated in nitric acid. The exposure panels, each 14 in. long and 4 in. wide, were prepared by the cooperating manufacturers. The shot-welded panels, type A, (Fig. 1) were assembled from three sections, each with an overlap of 1½ in. on which was a double row of four welds ¾ in. apart. The faying surfaces were usually protected with petrolatum pastes containing aluminum or copper powders. On type B panels having dissimilar metals in contact, the main sheet was sandwiched between the two dissimilar alloy strips, which were 1 by 4 in., and were joined to it by four rivets. The ratio of the area of the main sheet to the strips was 7:1 for each combination.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

[1] Metallurgist, National Bureau of Standards, Washington, D. C. (Now deceased.)

[2] Willard Mutchler, "Marine Exposure Tests on Stainless Steel Sheet," Technical Note No. 1095, Nat. Advisory Committee for Aeronautics, 1946.

The National Bureau of Standards exposure racks are located at the U. S. Naval Air Station, Hampton Roads, Va., an area representative of a temperate climate with marine conditions. While the present tests were in progress, it was twice necessary to change the site of the racks within the Naval Station, owing to wartime building expansions. During the first 2½ yr. of exposure, from June, 1938, to October, 1940, the racks were situated in an inlet of semi-brackish water named Boush Creek. The weather racks were directly over the water (Fig. 2(*a*)), with the panels 6 to 11 ft. above mean tide level, and suspended at an angle of 45 deg. During periods of storm or high winds the under

TABLE I.—PHYSICAL AND CHEMICAL CHARACTERISTICS OF THE STEEL SHEETS.

Designation[a]	Thickness, in.	Surface Finish[b]	Exposure[c]	Chemical Composition, per cent							
				Chromium	Nickel	Carbon	Manganese	Silicon	Sulfur	Phosphorus	Other Elements
A-1	0.017	2-B	1	19.99	9.82	0.09	0.49	0.271	0.010	0.019	....
A-5[d]	0.016	4	2	18.72	8.68	0.06	0.54[g]	0.36[g]	0.008[g]	0.018[g]	....
A-9	0.020	2-B	2	17.82	8.25	0.118	0.52	0.39	0.014	0.017	....
A-14	0.021	2-B	1	17.5	7.35	0.11	0.59	0.38	....	....	....
A-15	0.060	2-B	4	17.48	8.28	0.10	0.50	....	....	....	....
B-1	0.018	2-B	1	17.91	11.08	0.08	1.41	0.364	0.006	0.015	Mo 3.67
B-2	0.051	2-B	3	18.80	13.70	0.07	1.68[g]	0.29[g]	0.014[g]	0.008[g]	Mo 3.60
B-3[e]	0.023	2-B	2	19.00	13.74	0.05	1.52	0.60	....	0.008	Mo 3.40
B-7	0.063	2-B	3	17.79	10.72	0.05	1.27[g]	0.34[g]	0.012[g]	0.011[g]	Mo 2.70[g]
C-1	0.018	2-B	1	17.56	9.12	0.07	0.41	0.463	0.008	0.015	Ti 0.50
C-5	0.2	....	5	19.09	9.15	0.05	0.39	....	0.015	0.010	Ti 0.29
D-1[f]	0.031	2-B	1	18.40	8.56	0.08	0.50	0.47	0.020[g]	0.010[g]	Cb 0.79
D-5	0.018	2-B	1	17.84	9.90	0.08	0.46	0.200	0.007	0.015	Cb 0.53
E	0.018	1	1	17.70	1.62	0.08	0.72	0.518	0.021	0.012	....
F	0.063	2-B	4	18.3	4.1	0.07	3.95	....	....	....	....

[a] The various steels were prepared and supplied by cooperating firms, namely, the American Steel and Wire Co., the Carnegie-Illinois Steel Corp., The International Nickel Co., the Sharon Steel Corp., the Republic Steel Corp., and the Edward G. Budd Manufacturing Co., which also cooperated by preparing most of the shot-welded panels.

[b] These commercial finish designations signify: (1) pickled, (2-B) bright cold-rolled, (4) standard polish (architectural), ground (6) tampico brush, and (7) high luster. Finishes 6 and 7 were applied to a steel having essentially the same chemical composition as steel A-9. The steels were all passivated by immersion in 20 per cent nitric acid at about 60 C. for 30 to 60 min.

[c]
1. Exposed to tidewater and weather at Hampton Roads, Va. in June, 1938. Withdrawals made from weather racks after 7½, 24, and 36 months; from tidewater racks after 7½, 12, 24, and 36 months of exposure.
2. Exposed simultaneously to weather and sea water at Hampton Roads, Va., Kure Beach, N. C., and Chapman Field, Fla., in October and November, 1940. Withdrawals made after 6 months at all localities, and after 12 months at Hampton Roads and Kure Beach.
3. Exposed to tidewater and weather in June, 1938, at Hampton Roads and removed after 36 months.
4. Exposed to weather at Kure Beach in November, 1941, and withdrawn after 12 months.
5. Exposed to weather at Hampton Roads in May, 1942, and withdrawn after 12 and 36 months, in the form of R. R. Moore fatigue specimens, cut from 5/8-in. rod. Specimens were successively polished on 1/0, 2/0, 3/0, and 4/0 emery paper and then were passivated for 1 hr. in a 20 per cent solution of nitric acid, by volume, at 60 C.

[d] Annealed; ultimate strength, 100,000 psi.; other steels, unless otherwise indicated, were cold-rolled, with tensile strengths from 150,000 to 200,000 psi.

[e] In the ¼-hard condition; ultimate strength, 122,000 psi.

[f] Heat-aged, probably from 8 to 72 hr. at 440 F., to an ultimate strength of 180,000 psi.

[g] Ladle analyses; all others represent the manufacturer's check analysis on the billets.

surfaces occasionally were wetted with spray. A branch railway, about $\frac{1}{8}$ mile distant, caused light deposits of soot on the skyward surfaces. The tidewater exposure racks adjoined the weathering unit. The panels in tidewater were suspended vertically in the middle of the tide range, so that they were immersed at high tide and out of water at low tide for approximately 5-hr. periods twice daily.

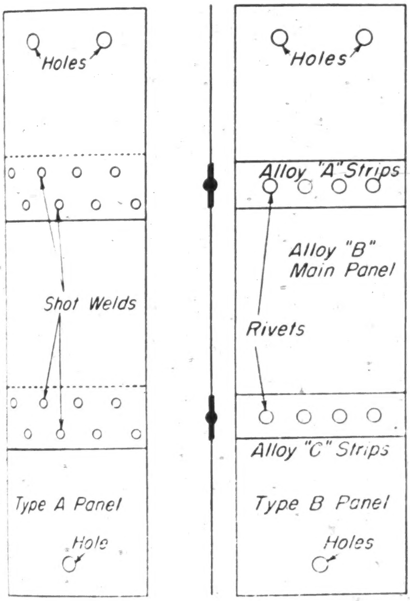

FIG. 1.—Types of Panels Used for Determining the Corrosion of Shot-welds, or of Dissimilar Metals in Contact.

The over-all dimensions of the panels were 4 by 14 in. The type A panels were shot-welded and the type B panels had the dissimilar metal strips riveted to the large sheet.

In 1940 the racks were moved to a lagoon (Fig. 2(*c*)), where the weather unit was on land, approximately 25 ft. from the sea water, and sheltered somewhat by a high earth embankment 50 ft. to the rear. The lagoon was a small inlet, near the mouth of Chesapeake Bay. It was at this period that some panels were simultaneously exposed at Chapman Field, Fla., with the cooperation of the Army Air Forces, and at Kure Beach, N. C. (Fig. 2(*b*)) with the cooperation of The International Nickel Co. The weather racks at these stations were located 25 to 40 ft. inland, facing the ocean.

METHODS OF EVALUATING CORROSION

The methods most frequently used to evaluate the corrosion consisted of (1) visual examinations, and (2) determination of the approximate fatigue limits on the steels before corrosion and after exposure for different lengths of time. The latter method, previously described,[3] entailed the use of flexural fatigue testing machines of the fixed deflection (constant strain) type, developed by G. N. Krouse for sheet (Fig. 3). Twelve specimens for test were cut from each panel in the direction of rolling. The value selected for the fatigue limit was the maximum stress at which no failure occurred in at least two "runs" of 10,000,000 cycles or more. The machines operated at approximately 2,500,000 cycles in 24 hr., and the fatigue failures on most of the corroded specimens occurred in much less than 2,000,000 cycles.

Tension tests were used to a limited extent for evaluating the corrosion, as was also a method entailing the preparation of a plastic replica of the surface, developed at the National Bureau of Standards,[4] to determine the degree of surface pitting.

RESULTS OF TESTS

Effect of Periodic Cleaning:

The panels in the exposure racks at Hampton Roads were inspected approxi-

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[3] W. H. Mutchler and J. A. Kies, "Fatigue Tests as a Means of Evaluating Corrosion of Sheet Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 42, p. 568 (1942).

[4] H. K. Herschman, Evaluation of the Finish of a Metal Surface by a Replica Method," *Journal of Research*, Nat. Bureau Standards, Vol. 34, p. 25 (1945) (*RP* 1625); *Mechanical Engineering*, Vol. 67, p. 119 (1945).

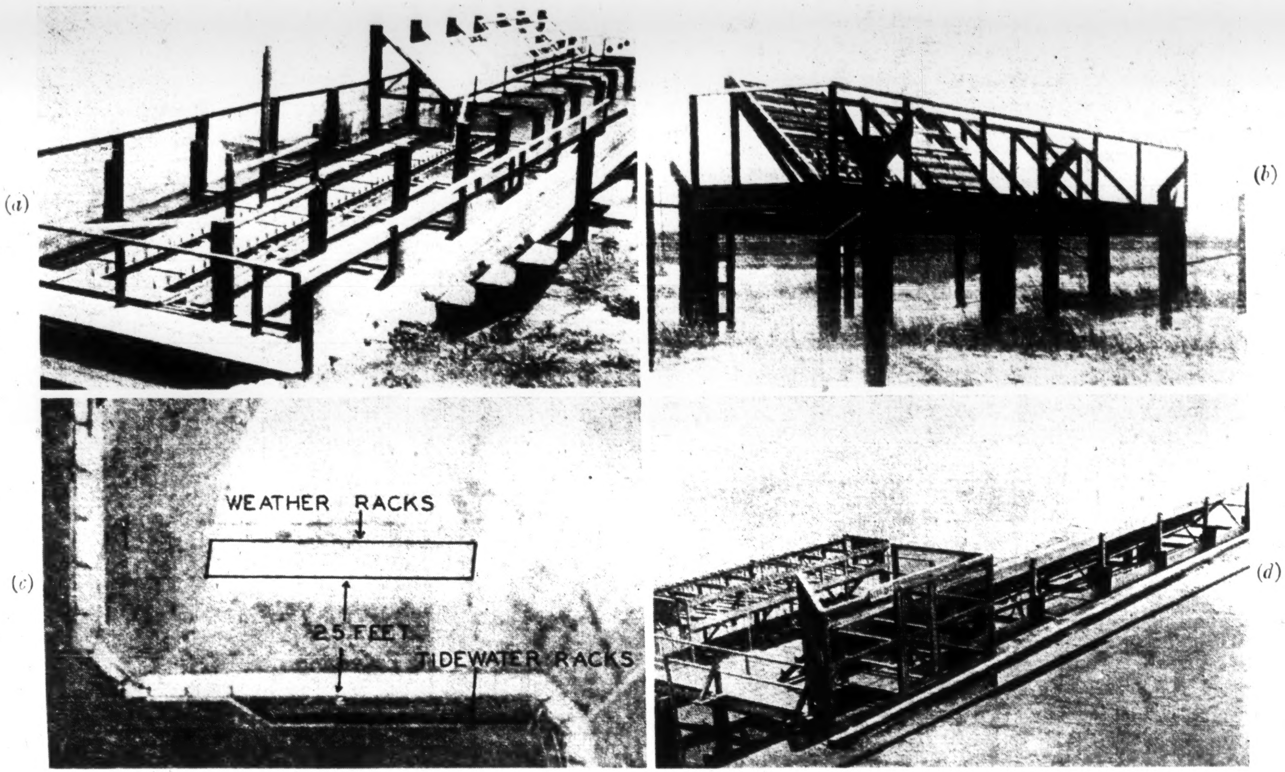
(a)
(b)
(c)
(d)
WEATHER RACKS
25 FEET
TIDEWATER RACKS

FIG. 2.—The Exposure Racks Used in the Investigation.

(a)—Weather and tidewater racks in Boush Creek, Hampton Roads, Va.
(b)—Weather racks on Kure Beach, Cape Fear, N. C.
(c)—Air view showing the relative location of the weather and tidewater racks in an artificial lagoon at Hampton Roads, Va.
(d)—The present exposure racks at Hampton Roads.

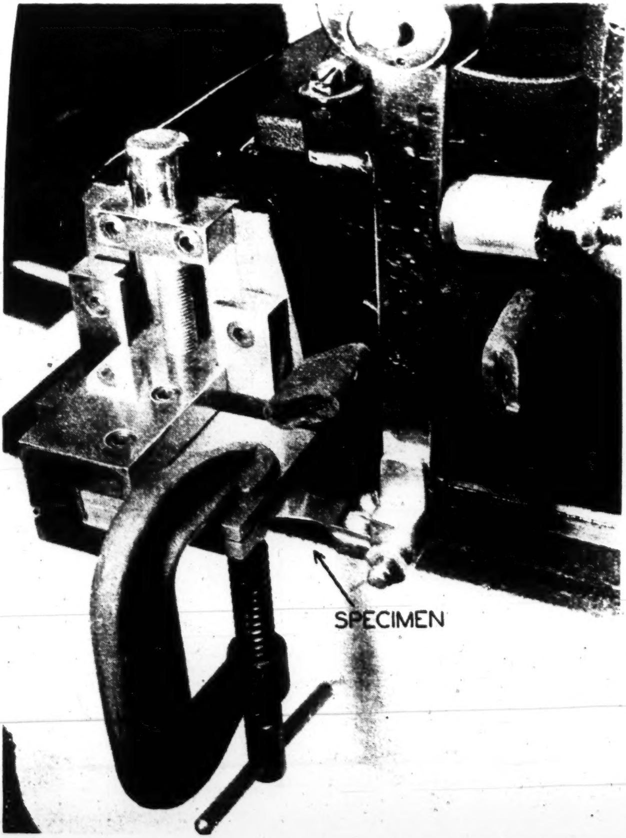

FIG. 3.—A Specimen in the Krouse Flexural Fatigue Testing Machine Showing the Method of Attaching It at the Fixed and Loading Ends.

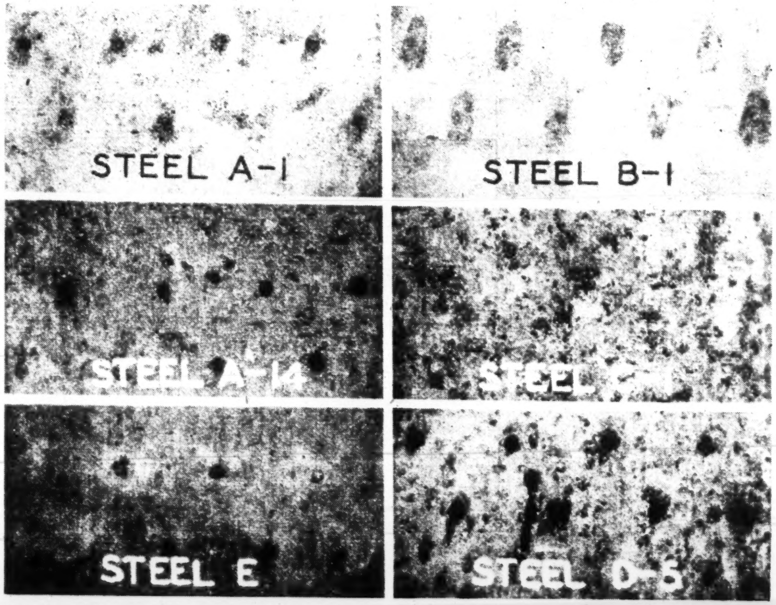
STEEL A-1
STEEL B-1
STEEL A-14
STEEL
STEEL E
STEEL D-5

FIG. 4.—The Earthward Surfaces of the Steel Panels Exposed to the Weather at Hampton Roads for 36 Months.

Steel B-1, containing 3.7 per cent of molybdenum, exhibited the least rust, while steel E showed the most rust. Steel A-1 was cleaned periodically, the others were not (× 1/2).

mately every 3 months. Areas of thin superficial, but adherent, rust usually appeared during the first month of exposure on the weather specimens. Within a few months, the rust covered the panels more or less uniformly and gradually became thicker with prolonged exposure. The rust was always present in greater quantity on the earthward surfaces. At the end of the third year, the rust was still relatively thin on the panels with initially polished finishes, and could be removed by the application of a suitable metal cleaner and polisher. Panels exposed in the tidewater seldom rusted.

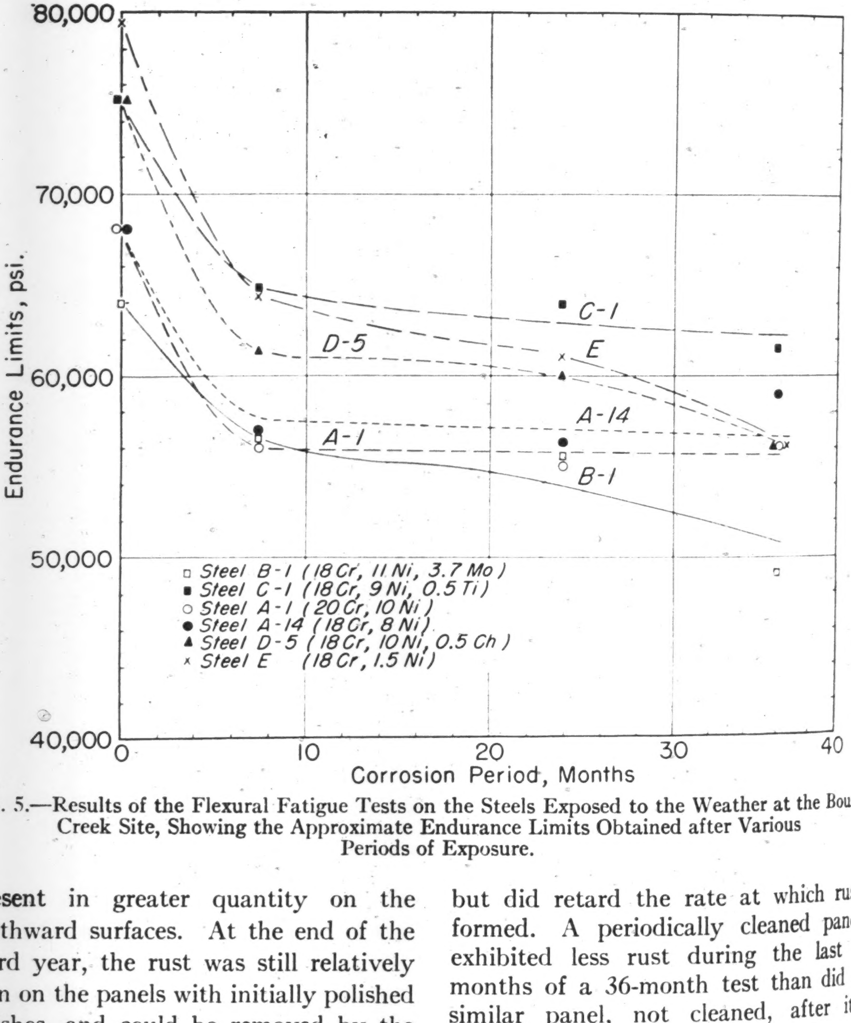

FIG. 5.—Results of the Flexural Fatigue Tests on the Steels Exposed to the Weather at the Boush Creek Site, Showing the Approximate Endurance Limits Obtained after Various Periods of Exposure.

At intervals of approximately 6 months, some of the panels were polished to remove the rust with a paste-type cleaner containing a grit, which left a water-repellent wax film on the metal. Minute pits were observable under most of the rusted areas after such cleaning. The cleaning did not prevent corrosion, but did retard the rate at which rust formed. A periodically cleaned panel exhibited less rust during the last 6 months of a 36-month test than did a similar panel, not cleaned, after its initial 6 months of exposure.

Effect of Chemical Composition:

The panels initially exposed at Hampton Roads in 1938 (Table I, footnote *c*, section 1) included the following:

Steel	Nominal Composition, per cent		
	Chromium	Nickel	Other Elements
A-1	20	10	....
A-14	17.5	7.5	....
B-1	18.0	11.0	Mo 3.7
C-1	17.5	9.0	Ti 0.5
D-1	18.0	8.5	Cb 0.8
D-5	18.0	10.0	Cb 0.5
E	18.0	1.5	....

The inspections of these panels consistently revealed that steel B-1, containing molybdenum, was very much less rusted than the others (Fig. 4). Steel E, which had a pickled surface finish, exhibited the most rust. The others were intermediate in appearance and too similar to permit of reliable differentiation.

The following data, obtained by the plastic replica method,[4] agree well with the results from other methods and indicate that this procedure may prove of value in determining the degree of surface roughness and pitting. The higher the values, given in millivolts $\times 10^{-1}$, the greater the surface roughness.

Steel	Uncorroded	Weathered 36 months at Hampton Roads	Increase
A-1	6.7	41.0	34.3
D-5	8.5	23.2	14.7
B-1	12.2	15.2	3.0

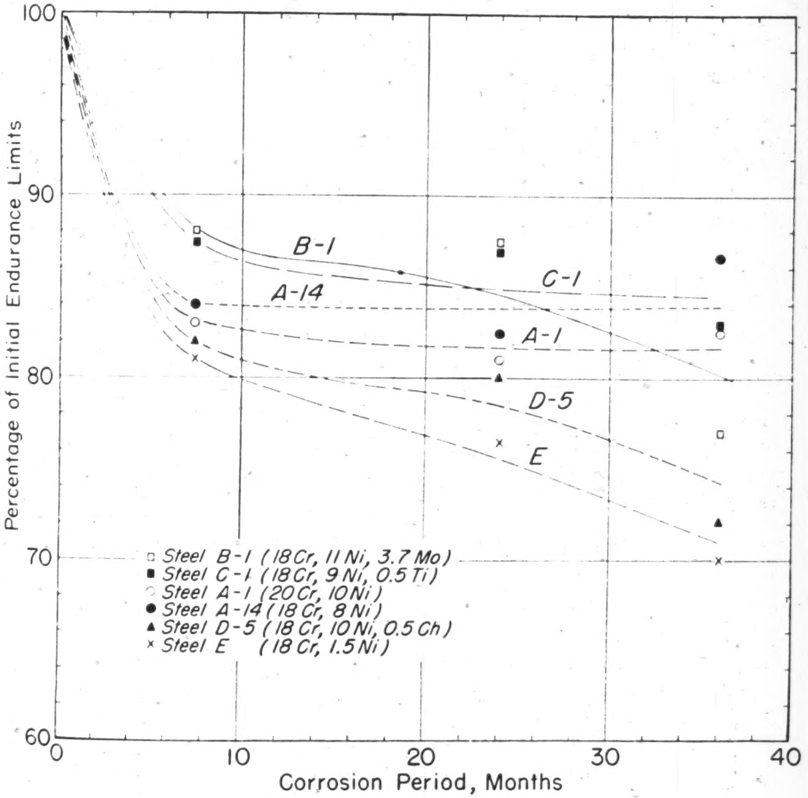

FIG. 6.—The Data from Fig. 5, Plotted on a Percentage Basis, to Permit Better Comparisons of the Rates of Corrosion for the Steels Exposed to the Weather at Boush Creek.

The data from the fatigue tests are plotted to reveal the approximate endurance limits (Fig. 5) and to permit a direct comparison of the alloys of different chemical compositions (Fig. 6). These data reveal that most of the corrosion occurred during the first 6 months of exposure and that thereafter the rate of attack was much slower. Although the steels containing additions of molybdenum and titanium are shown to be somewhat more resistant to corrosion than the others, it should be noted that the average variation in the percentage of loss in the fatigue limits was only approximately ± 3 per cent from the mean value.

TABLE II.—COMPARISON OF THE TENSILE PROPERTIES WITH THE ENDURANCE LIMITS OF THE STEEL SHEETS EXPOSED AT KURE BEACH, N. C., AND CHAPMAN FIELD, FLA.

Steel	Locality	Exposure Period, months	Tensile Properties			Endurance Limit, psi.
			Tensile Strength, psi.	Yield Strength, psi.	Elongation in 2 in., per cent	
A-15	Not exposed	0	166 300	128 000	22.0	69 000
	Kure Beach, weather	12	166 000	129 000	22.0	65 000
F	Not exposed	0	190 000	124 900	27.5	75 000
	Kure Beach, weather	12	190 000	127 000	27.0	74 000
A-5	Not exposed	0	98 500	46 400	64.3	51 000
	Chapman Field, weather	6	97 600	45 800	64.8	46 400
	Chapman Field, tidewater	6	98 500	46 300	65.8	44 000
A-9	Not exposed	0	188 400	149 400	6.6	74 000
	Chapman Field, tidewater	6	188 200	144 100	6.1	74 000
B-3	Not exposed	0	121 600	109 200	19.8	41 000
	Chapman Field, weather	6	124 600	110 700	17.6	35 000
	Chapman Field, tidewater	6	124 400	110 200	17.7	32 500

When it became apparent that the greatest loss in fatigue limit occurred during the first 6 months of exposure, it was thought that such behavior might be characteristic only for sheet. A few $\frac{1}{8}$-in. diameter R. R. Moore fatigue specimens (Table I, footnote *c*, section 5) were therefore also exposed to the weather. This alloy, steel C-5, had an initial endurance limit of 75,000 psi. After 1 yr. of exposure a value of less than 55,000 psi. was indicated, while after 3 yr. a value of 52,000 psi. was obtained. The rate of loss in endurance limit was therefore of the same order as for the sheet specimens.

On a few samples only, a direct comparison was made between the loss in tensile properties and the loss in endurance limits resulting from corrosion. Two steels, tested cooperatively with The International Nickel Co., and designated A-15 and F (Table I, footnote *c*, section 4) were exposed to the weather for 1 yr. at Kure Beach, N. C., approximately 250 yd. from the ocean beach, and facing south at an angle of 30 deg. The fatigue test data (Table II) indicated that steel F, containing 18 per cent chromium, and 4 per cent each of manganese and nickel, was somewhat more resistant than the ordinary 18-8 steel A-15. Tension tests on these panels, and on others exposed at Chapman Field (Table II), failed to reveal any significant difference. The differentiation shown by the fatigue tests was attributed to their greater sensitivity to notch effects, as exemplified by shallow surface pits.

One half of a corrosion-resistant steel airplane wing is currently being exposed to the weather at Hampton Roads, suspended in the exposure racks about 8 ft. above the mean tide (Fig. 2(*d*)). After 6 months of exposure, the upper and lower outer surfaces were superficially rusted, and were similar in appearance to the individual panels already described. On the partially sheltered lower inner wing surface, heavy red rust extended inward from 6 to 10 in. from the cut edge. Rust deposits on the outer surfaces were heaviest on the bottom side, and, in general, on the shot welds.

Corrosion on Shot-Welds and at Faying Surfaces:

On panels exposed to the weather at Hampton Roads, there was a marked tendency toward slightly heavier deposits of rust on the shot-welds than on the rest of the sheet. Welds on the molybdenum-containing steels were the least rusted, while those on steel E were the most rusted.

Tension tests, made on each of the 16 welds on each panel, revealed that only a few of the welds showed marked losses in breaking loads or exhibited evidence of severe corrosion, after the maximum exposure period of 36 months. These failures were probably related to conditions during the welding operation, rather than to the inherent chemical characteristics of the sheets. The breaking loads for the individual welds on steel E ranged from 625 to 675 lb., and from 480 to 600 lb. for the other steels. The welds were approximately $\frac{1}{8}$ in. in diameter. The welds on steel E, however, withstood a twist of only 10 deg., while those on the austenitic type of alloys withstood a twist of approximately 90 deg. before failure.

On most of the steels, petrolatum grease was applied at the faying surfaces prior to shot-welding, and little or no corrosion was noted at the overlaps. After 3 yr. of exposure to the weather at Hampton Roads, most of the grease was still *in situ*. Aluminum or copper powders impregnated in the grease afforded little, if any, added protection. When grease was omitted, however, from 50 to 80 per cent of the faying surfaces was covered with rust, considerably thicker than that which appeared on the outer surfaces of the sheet. On steel E some rusting occurred at the faying surfaces, even though grease had been applied.

Effect of Surface Treatments and Finishes:

Steels B-2 and B-7 (Table I, footnote *c*, section 3) containing 3.6 and 2.7 per cent of molybdenum, respectively, were exposed at Hampton Roads with the two-fold objective of determining the effect of the difference in molybdenum content and of various surface treatments. Periodic visual inspections throughout the 3-yr. exposure indicated that the steel with the lower molybdenum rusted somewhat more rapidly. The difference was adjudged immaterial for most practical applications, and the rust on both steels was less than on those of the other compositions investigated.

These steels were exposed to the weather in the following conditions of surface treatment: (1) passivated 60 min. in 20 per cent nitric acid at 60 C.; (2) pickled 20 to 30 min. in a solution containing 20 per cent of nitric acid and 4 per cent of hydrofluoric acid, by volume, at 60 C., and passivated; (3) passivated, pre-surfaced 30 min. in a 10 per cent ferric chloride solution at room temperature; (4) pickled, passivated, pre-surfaced, and passivated. The panels subjected to treatment 2 exhibited the least rust while those treated by method 4 were rated second by visual examinations. Panels surface-treated under conditions 1 and 3 were quite similar to each other in appearance, but were inferior to those given the pre-pickling treatment.

When the exposure racks were relocated in the lagoon at Hampton

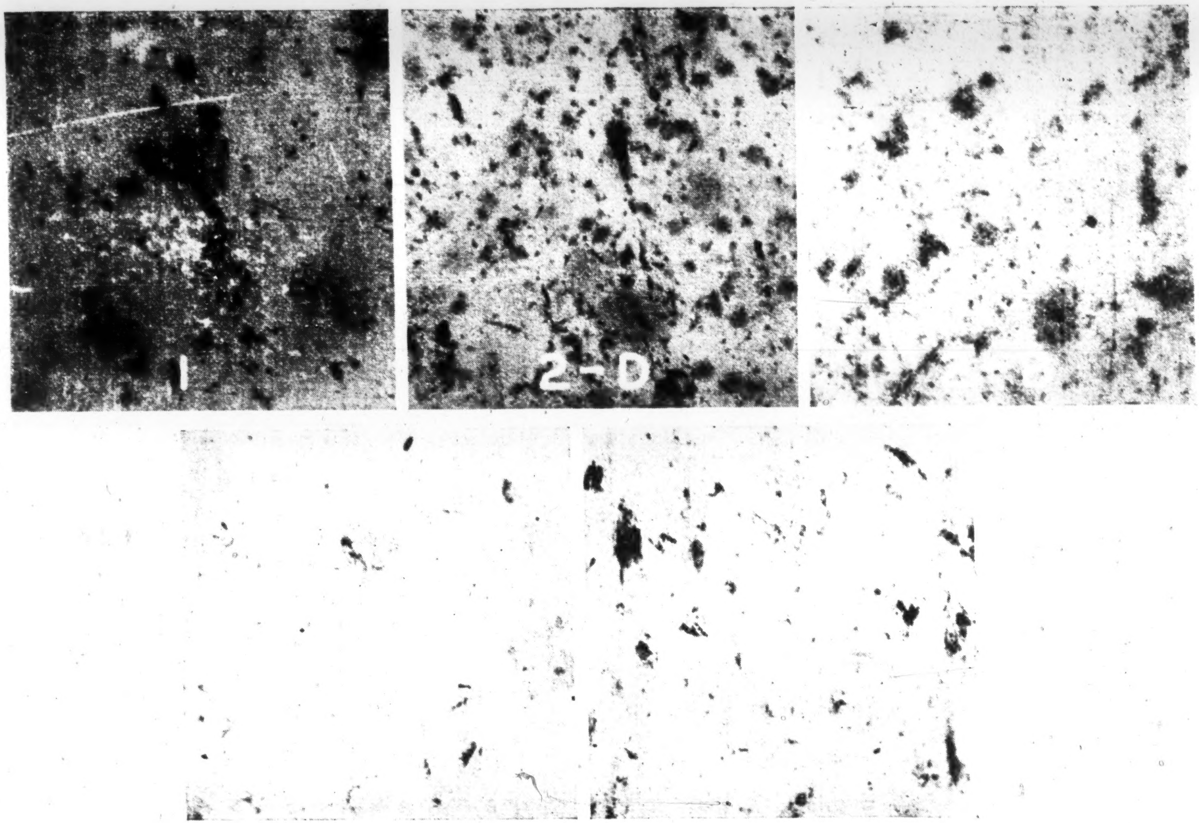

FIG. 7.—The Earthward Surfaces of Panels with the Indicated Commercial Surface Finishes, after Exposure to the Weather at Hampton Roads for 6 Months.

The rust tended to decrease in quantity and size as the degree of surface polish improved (× 1).

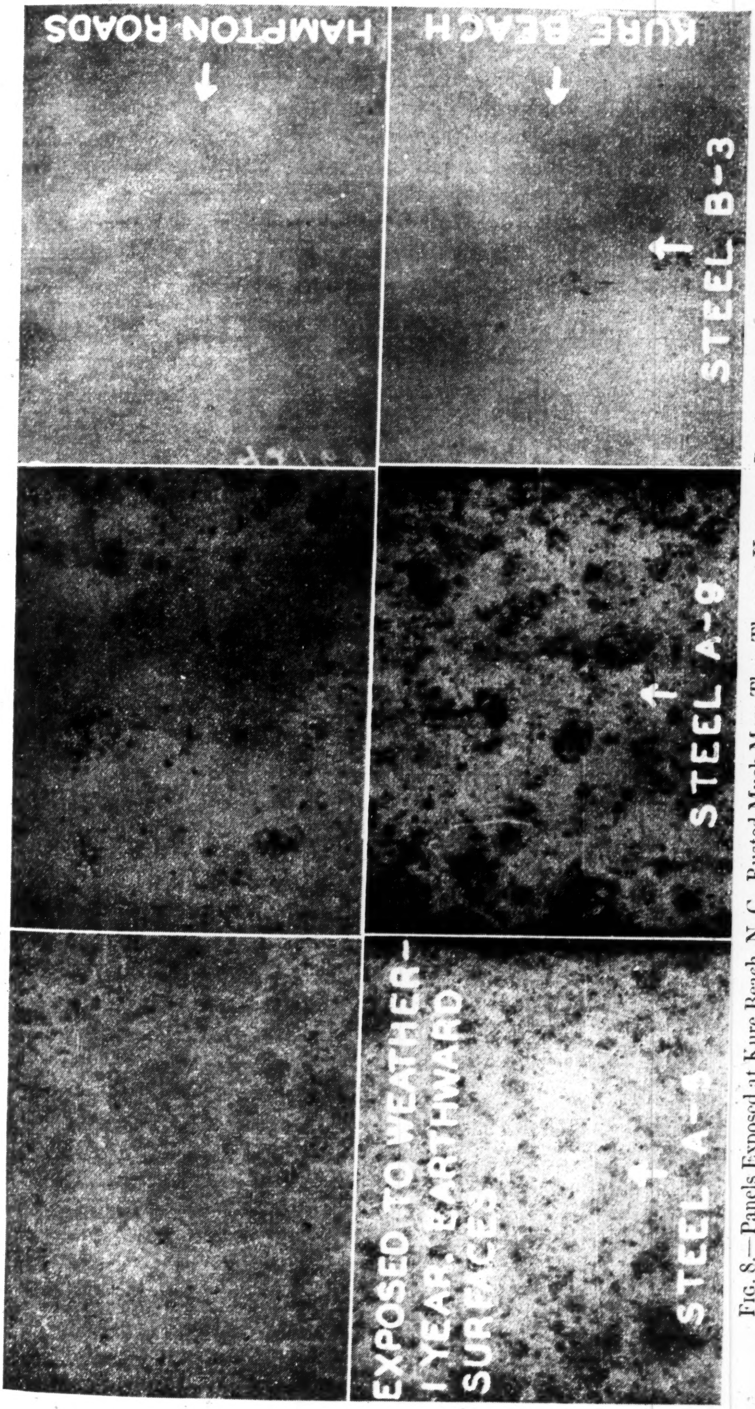
HAMPTON ROADS
KURE BEACH
STEEL B-3
STEEL A-9
STEEL A-1
EXPOSED TO WEATHER—
1 YEAR, EARTHWARD
SURFACES

FIG. 8.—Panels Exposed at Kure Beach, N. C., Rusted Much More Than Those at Hampton Roads, Va., or at Chapman Field, Fla.

At each locality the molybdenum-containing steel was the least rusted (× 3/5).

Roads, a series of unwelded panels approximately duplicating those of Table I, footnote *c*, section 1, were inserted. These steels, however, had a number of different commercial degrees of surface finish. Panels having the bright-rolled finish (2-B) rusted at a slightly slower rate than those initially exposed at the Boush Creek site, probably owing in part to the greater distance from the water at the lagoon. The quantities of rust on the steels of different compositions, however, remained in the same relative order.

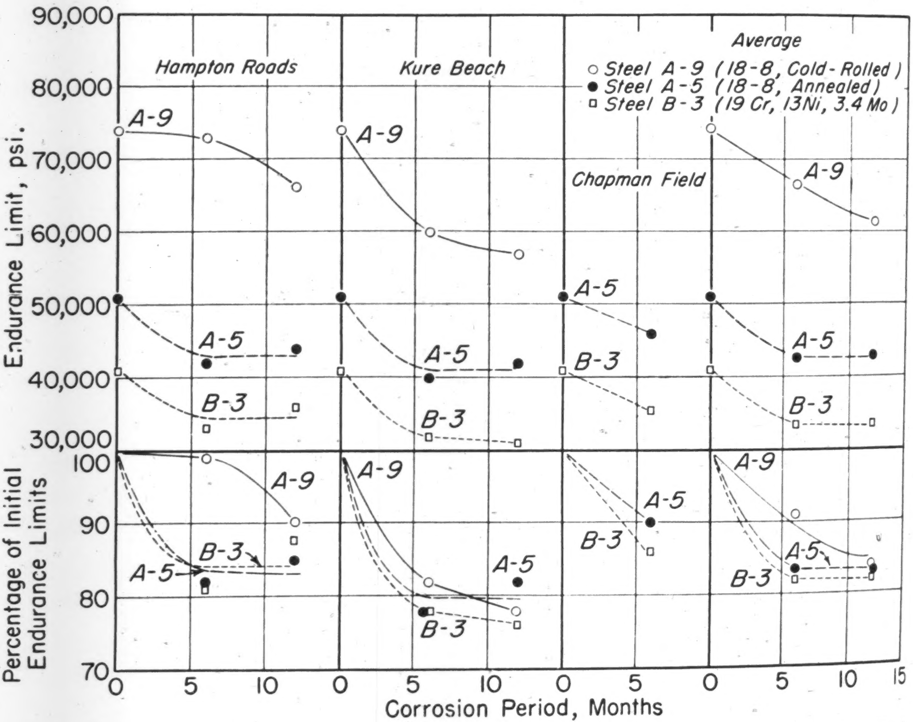

FIG. 9.—Results of the Fatigue Tests on Each Steel Exposed to the Weather Simultaneously at Each of Three Localities.

The amount of rust on panels of a given composition varied with the surface finish. The duller finishes, that is, the pickled (1)[5] and the dull cold-rolled (2-D), acquired noticeably thicker rust deposits than the others, the individual areas attaining diameters of approximately $\frac{1}{2}$ in. The rust on the bright-rolled (finish 2-B),[5] ground architectural (4),[5] and the standard polish, satin, tampico brush finish (6) was much thinner, and the individual areas tended to be smaller, seldom exceeding $\frac{1}{8}$-in. in diameter (Fig. 7). Panels with a mirror-like finish (7), made by grit-grinding the bright-rolled (2-B) surfaces with 320 emery, and buffing to a high luster, exhibited the least rust. Certain of these panels were periodically cleaned. The cleaner usually removed the rust only partially from the dull finished panels, after vigorous rubbing. Rust could be entirely removed, without the application of much pressure on finishes 2-B, 4, and 6, and was readily removed with even lighter rubbing on the buffed finish 7.

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[5] See Table I, footnote *b*.

Effect of Locality of Exposure:

The panels exposed simultaneously at Hampton Roads, Va. (at the lagoon), Kure Beach, N. C., and Chapman Field, Fla., in 1940, consisted of three steels (Table I, footnote *c*, section 2). Steel A-5 was of the straight 18-8 type, annealed, with a ground architectural surface finish, and a tensile strength of approximately 100,000 psi. Steel A-9 was also of the straight 18-8 type, bright cold-rolled, with a tensile strength of 188,000 psi. Steel B-3 contained 3.4 per cent of molybdenum, and was bright cold-rolled $\frac{1}{4}$-hard, with a tensile strength of 122,000 psi.

The samples exposed at Kure Beach were rusted much worse than those at the other two locations (Fig. 8). The molybdenum-containing steel was the least rusted at all locations. Although steels A-5 and A-9 were very similar in appearance at each place, the annealed steel A-5 seemed to be slightly the more rusted. The three steels, however, showed approximately the same loss in fatigue properties at Kure Beach (Fig. 9), but at Hampton Roads, steel A-9 showed somewhat less loss than the others.

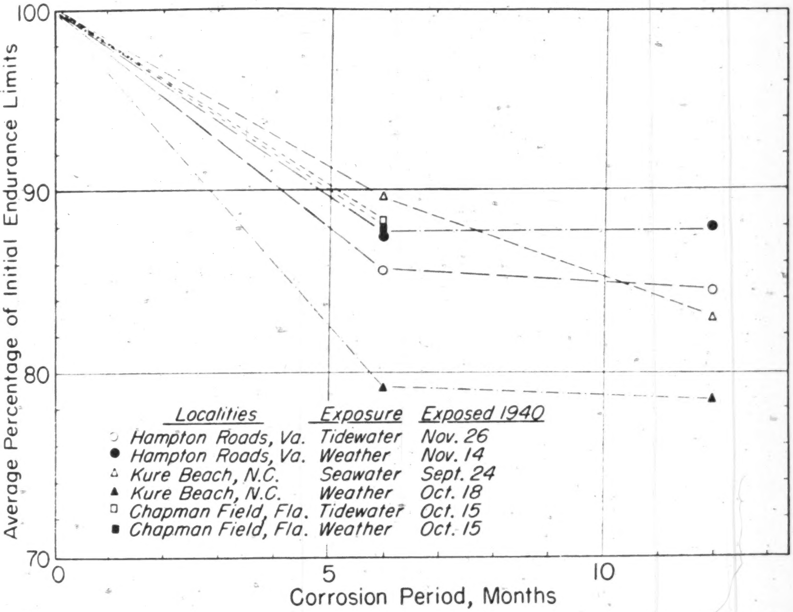

FIG. 10.—The Average Percentage of Loss of Endurance Limit Is Shown to be Very Similar for Steels A-5, A-9, and B-3 Exposed to the Weather or Tidewater Simultaneously at Hampton Roads, Va., Kure Beach, N. C., and Chapman Field, Fla.

The average deviation was less than ±3 per cent for most of the conditions of exposure, but panels exposed to the weather at Kure Beach consistently exhibited the greatest loss.

The average data for the three steels, plotted on the basis of the percentage loss of the initial endurance limit (Fig. 10), reveal the comparative rates of corrosion at each locality. The panels exposed to the weather at Kure Beach showed greater loss than those exposed under any of the other marine conditions, including those continuously or intermittently immersed in the sea water. Minor changes in location at a given locality may be a determining factor (Fig. 11) as to whether corrosion is more severe on panels exposed to the weather, or on those exposed to tidewater.

Irrespective of whether panels were exposed to the weather or to sea water at the three localities, the variation in the fatigue limits for all the steels was usually less than ± 3 per cent for any specified period of exposure. This indicates that, for most practical applications, these steels may all be expected to exhibit markedly similar resistance to corrosion. The relatively narrow range of the loss in fatigue-limit values for most of the steels tested also indicates that the order of susceptibility of each steel to corrosion may be expected to vary, within the ranges established, for different heats of metal exposed under the same or comparable conditions.

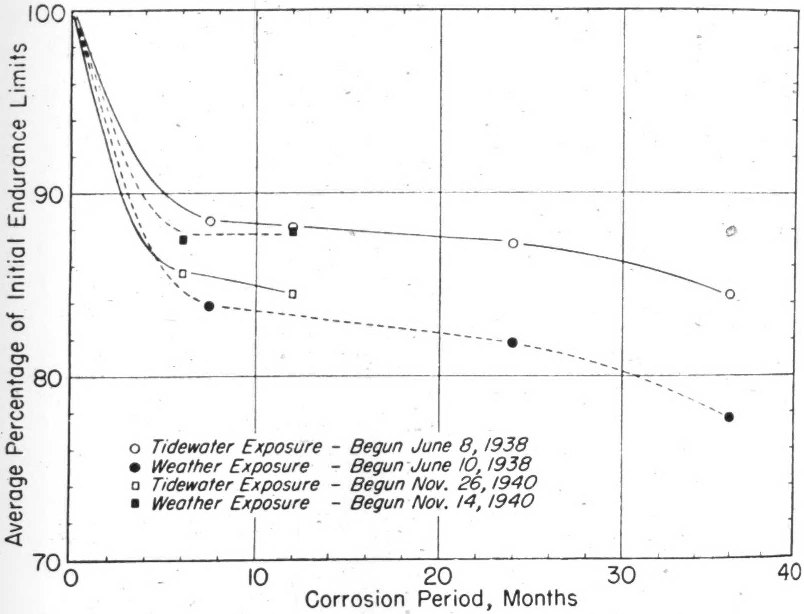

FIG. 11.—**Panels Exposed in 1938 Were Located at the Boush Creek Site, While Those Exposed in 1940 Were Located at the Lagoon, at Hampton Roads, Va.**

The data are the average for all the steel panels, irrespective of their chemical compositions. The average deviation is less than ±3 per cent. The panels exposed directly over the sea water at Boush Creek showed greater loss in endurance limits than those exposed to the tidewater. At the lagoon site, where the panels were exposed on shore, the tidewater panels exhibited the most loss.

Effect of Contact with Light Metal Alloys:

Steel C-1, stabilized with 0.5 per cent of titanium, was used on the panels having corrosion-resistant steel exposed in contact with aluminum or magnesium alloys at Hampton Roads. The light metals investigated included the following aluminum and magnesium alloys:

Alloy	Nominal Composition, per cent						
	Mg	Cu	Mn	Si	Zn	Cr	Al
24S-T[a]	1.5	4.5	0.6	...	...	...	bal.
53S-T	1.3	...	...	0.7	...	0.25	bal.
52S-½H	2.5	...	...	...	...	0.25	bal.
Dowmetal M	bal.	...	1.5	...	...	...	...
Dowmetal H	bal.	...	0.2	...	3.0	...	6.0

[a] Also as Alclad 24S-T, coated on both sides with aluminum.

The light alloys all proved highly anodic to steel C-1, with the magnesium

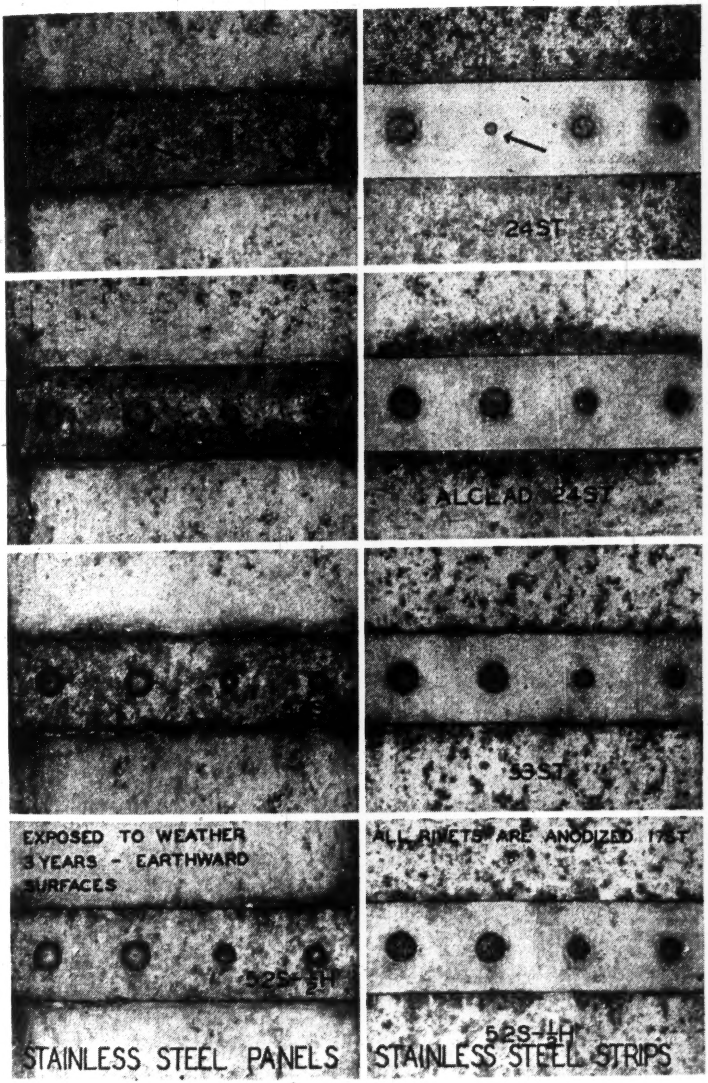
24ST
ALCLAD 24ST
53ST
EXPOSED TO WEATHER
3 YEARS - EARTHWARD
SURFACES
ALL RIVETS ARE ANODIZED 17ST
52S-½H
52S-½H
STAINLESS STEEL PANELS
STAINLESS STEEL STRIPS

FIG. 12.—Earthward Surfaces of Corrosion-Resistant Steel Coupled with Aluminum Alloys, and Exposed to the Weather at Hampton Roads for the Periods Indicated.

Note that rivet heads (arrow) joining the steel to alloy 24S-T occasionally broke off because of the stresses imposed by corrosion products at the faying surfaces (× ½).

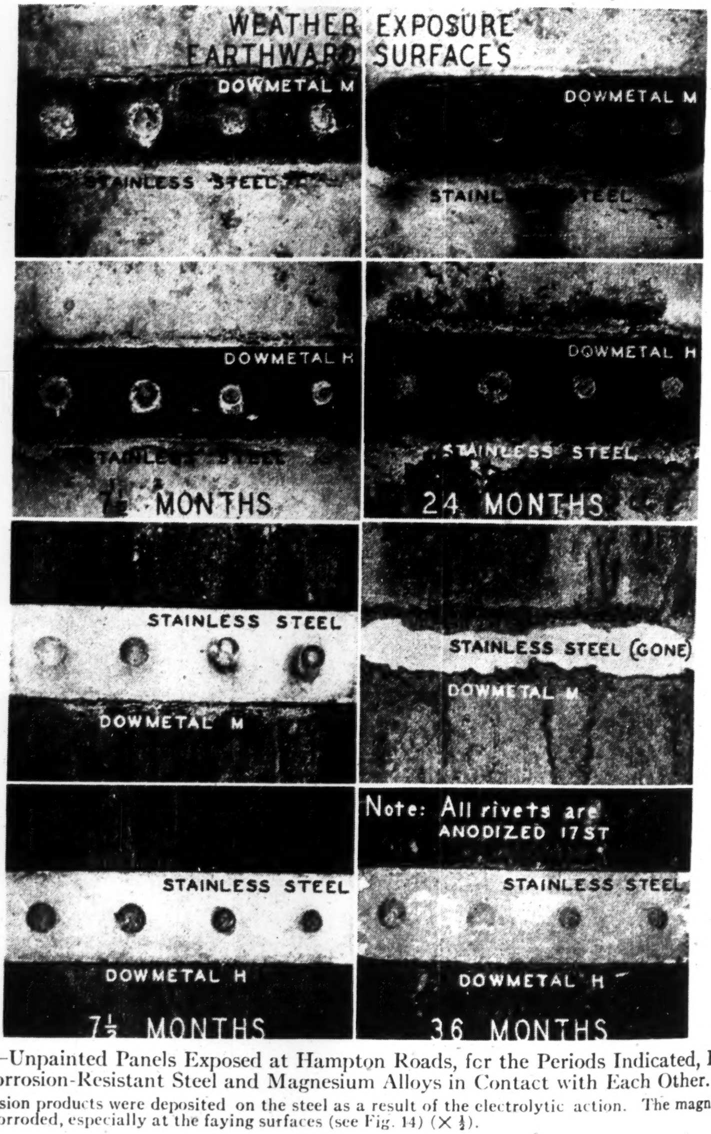
WEATHER EXPOSURE
EARTHWARD SURFACES
DOWMETAL M
STAINLESS STEEL
DOWMETAL M
STAINLESS STEEL
DOWMETAL H
STAINLESS STEEL
7½ MONTHS
DOWMETAL H
STAINLESS STEEL
24 MONTHS
STAINLESS STEEL
DOWMETAL M
STAINLESS STEEL (GONE)
DOWMETAL M
Note: All rivets are
ANODIZED 17ST
STAINLESS STEEL
DOWMETAL H
7½ MONTHS
STAINLESS STEEL
DOWMETAL H
36 MONTHS

FIG. 13.—Unpainted Panels Exposed at Hampton Roads, for the Periods Indicated, Having Corrosion-Resistant Steel and Magnesium Alloys in Contact with Each Other.

White corrosion products were deposited on the steel as a result of the electrolytic action. The magnesium alloys were severely corroded, especially at the faying surfaces (see Fig. 14) (× ½).

alloys being selectively corroded more severely and more rapidly than the aluminum-base alloy members of the couples. The galvanic corrosion, of course, occurred very much faster in the tidewater racks than in the weather racks. The area relationships are also important, and corrosion on the light alloys was always much more severe when their areas were small compared with the steel.

Macroscopical and microscopical examinations revealed that the severity of the corrosion on the light alloys, when coupled with corrosion-resistant steel, decreased in this order: Dowmetal M, Dowmetal H, 24S-T, Alclad 24S-T, 53S-T, and 52S-$\frac{1}{2}$H. This arrangement does not indicate the order of the potential differences involved, since the individual alloys vary considerably in their inherent susceptibility to corrosion.

Corrosion products accumulated in abundance between the dissimilar surfaces in contact, and after the first year were sufficient to cause the heads of some rivets to break off and to cause stress-corrosion cracking on the 24S-T and Alclad 24S-T sheets. In a few instances, stress-corrosion cracks developed on the steel strips, joined to magnesium alloy main panels, at the circumference of the rivet heads (Fig. 14). When the light alloy main panels and the steel strips were painted, corrosion at the faying surfaces was usually delayed for from 6 to 12 months. Paint does not adhere well to polished corrosion-resistant steel and it failed on these samples within a few months on the exposed surfaces.

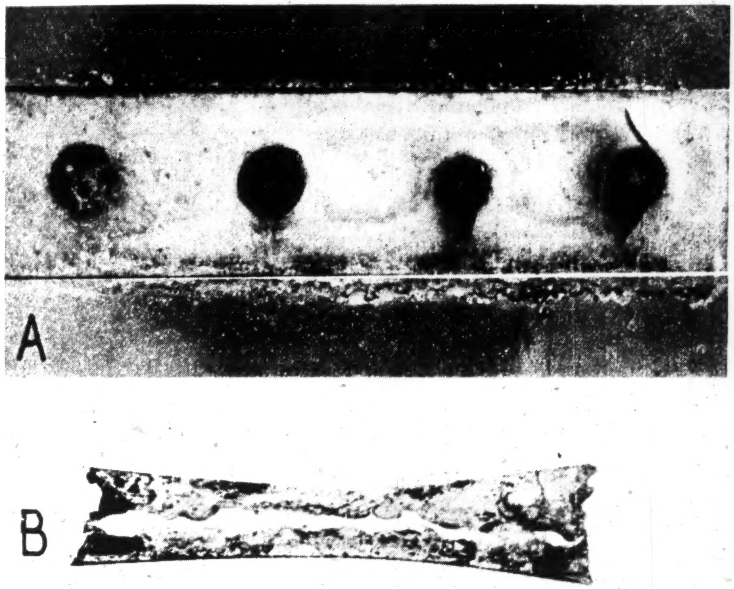

FIG. 14.—Example of Stress Corrosion on Panels Having Corrosion-Resistant Steel Coupled with Light Metal Alloys and Exposed to the Weather for 2 yr. at Hampton Roads.

(A) Crack on a steel strip joined to a magnesium alloy panel, Dowmetal M (× 1).

(B) Cross-section showing the large amount of corrosion products at the faying surfaces (× 2½).

DISCUSSION OF LABORATORY AND WEATHERING TESTS

The occurrence of corrosion in greater abundance on the under surfaces of specimens exposed to the weather is characteristic for most metals. No entirely satisfactory theory has yet been promulgated to account for this behavior. The surface appearance of specimens corroded by various laboratory methods is seldom the same as that obtained during weathering, particularly with respect to the size and distribution of the corrosion products. Subcommittee VII on Weather of A.S.T.M. Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys has, for one of its objectives, a more thorough study of the factors influencing outdoor corrosion, and the appended remarks may prove of interest in that connection.

A rather simple and purely qualitative experiment was made on a few metal sheets, including corrosion-resistant steel, in which the surface appearances obtained on weathering were duplicated. Metal strips, approximately 2 ft. long and from 1 to 3 in. wide, were bent rectangularly at each end. One end was placed in a beaker of boiling water, the other in a beaker of ice water, to assure a temperature gradient. The horizontally situated sheet was sprayed with a dilute solution of sodium chloride on its top and bottom surfaces, the spraying being repeated as soon as drying was complete. Corrosion products first formed, usually in a narrow band less than $\frac{1}{2}$ in. wide, at an area on the under side near, but some inches removed from, the hot end. With repeated sprayings and alternate dryings, the width of the band gradually increased toward the colder end, and finally attained an apparent maximum width which varied from 3 to 7 in. for the metals tested. On the more susceptible metals, after prolonged repetition of the spraying cycle, corrosion products eventually appeared rather uniformly distributed on the under side of the colder end.

This behavior is believed to be analogous to that which results in outdoor weathering. It suggests the presence of electrolytic cells, perhaps of the oxygen-concentration variety, which attain their maximum activity during the periods of retention of films of moisture possessing certain critical thicknesses and oxygen contents. The maximum activity appears to occur after the films become discontinuous and agglomerate into droplets, owing to surface tension. The length of time that the critical films are present probably determines the rate at which corrosion products form. This would indicate that in outdoor weathering, the rates of drying of the intermittently deposited moisture films, as determined by certain critical combinations of the temperature, sunlight, wind, and humidity, are the external factors of prime importance in causing corrosion. This may also explain why attempts to correlate corrosion with the absolute or average values of these factors, or of data related to the frequency and quantity of precipitations (rain, snow, dew, etc.) are usually unsuccessful. Externally the presence of other ions in the cell, such as chlorine or sulfur, peculiar to a specific atmosphere, represents another important variable in influencing the rate of corrosion.

The metallurgist, although he knows better, too often regards his exposure panel in the light of a homogeneous surface. The inherent nonhomogeneities of the metal surface, as well as the local differences in its ability to form protective surface films, and, eventually, the presence of the initial corrosion products, all combine to determine where and in what manner the corrosion takes place.

In view of the promising results obtained with the rather crude temperature gradient-repeated spray experiment, it is planned to construct an apparatus permitting a temperature gradient on the metal, in which temperature of the air, humidity, rate of drying, etc., are susceptible to control, and thus perhaps obtain some fundamental information regarding the critical combinations of each of these factors as related to the corrosion of different alloys.

SUMMARY

Corrosion-resistant steel sheets were exposed for approximately 3 yr., under extreme marine conditions, as exemplified by weather exposure in close proximity to, or immersed in, sea water.

Rust deposits usually formed in greatest quantity and thickness, especially on the under sides of the panels, within the first 6 to 12 months of weathering. Thereafter, significant changes in surface appearance did not ordinarily occur. Rust rarely formed on panels exposed to sea water, particularly those with bright-rolled, or higher, degrees of surface finish. In general, the better the degree of surface finish, the less was the tendency toward the formation of adherent rust, for sheets of the same chemical composition. Flexural fatigue tests likewise demonstrated that the greatest damage usually occurred during the first 6 months of exposure.

The 18-8 type steels, containing from 2.5 to 3.5 per cent molybdenum exhibited much less rust on weathering than the same type steels with or without additions of titanium or columbium. The fatigue tests revealed a slight superiority for the 18-8 type steels containing molybdenum and titanium. The relatively narrow range of the loss in fatigue limits (± 3 per cent of the mean values for most of the steels investigated) indicates that the order of susceptibility may be expected to vary, within the ranges established, on different heats of metal exposed under the same, or other, marine conditions.

The tests also revealed that at a given locality the damage resulting from exposure to the weather may, or may not, be worse than that resulting from exposure to sea water. Minor changes, such as the proximity of the weathering panels to the sea water, or the presence in the latter of certain sea organisms, may determine the severity of the corrosion.

Shot-welds were slightly more susceptible to the formation of rust, on panels exposed to the weather, than was the rest of the sheet. Welds on the steels containing molybdenum were the least rusted. The breaking loads of the welds usually were relatively unchanged after exposure for 3 yr. The most severe rusting of the welded sheets occurred at the faying surfaces. Applications of greases such as petrolatum, with or without aluminum or copper powders, were quite effective in preventing such corrosion.

Pickling, prior to passivating surface treatments, tended to improve the resistance of corrosion-resistant steels to corrosive attack. Pre-surfacing (treatment in ferric chloride) coupled with passivation was of doubtful merit, and surfaces so treated appeared to corrode slightly more than those which were passivated but not pre-pickled.

Aluminum and magnesium alloys, especially the latter, were highly anodic to corrosion-resistant steels, and were severely attacked when in contact with them. When the area of the steel is small compared with the light alloy, the minimum corrosion of the latter results from electrolysis. Suitable paints at the faying surfaces are adequate in preventing corrosion there for periods of at least 6 to 12 months of weathering under marine conditions.

DISCUSSION

MR. WILLIAM H. FINKELDEY.[1]—I understood Mr. Waldron[2] to say that all the corrosion-resistant steel specimens including those that were exposed in the atmosphere were passivated.

MR. LEO WALDRON.[3]—All were passivated except panels B-2 and B-7 which were treated with ferric chloride after passivation.

CHAIRMAN F. L. LAQUE.[4]—If the chair may be permitted an observation, on the basis of the large number of corrosion-resistant steels of different composition that we have exposed for 5 yr., the appearance of these specimens after exposure for 5 yr. was not much different from their appearance after 6 months. Apparently, the small amount of corrosion responsible for staining is self-arresting and decreases considerably in rate as the time of exposure is prolonged. This seems to be true, whether or not the specimen has been passivated immediately prior to exposure.

There is one other observation I should like to make, as a discusser rather than as chairman, with respect to the statement by Mr. Mutchler which he made in 1942 as well to the effect that damage to corrosion-resistant steel in a marine atmosphere as measured by fatigue is likely to be greater in the atmosphere than when immersed in sea water. I think that must be, to my way of thinking, a rather peculiar and not a normal behavior because exposure in sea water, where marine organisms can develop and can induce pitting, has produced on many hundreds of corrosion-resistant steel panels such intense pitting that I cannot conceive that fatigue tests on specimens that might include long pits, maybe an inch long, could possibly give a fatigue life of more than a few seconds.

I think it would be wise not to reach the general conclusion that corrosion-resistant steels are susceptible to more damage in a marine atmosphere than when immersed in sea water. I do not doubt but that the particular specimens gave the data Mr. Mutchler recorded, but I do not think the results should be applied too broadly.

MR. S. B. ASHKINAZY.[5]—I believe there is one significant point that can be brought out here and I should like to have the speaker note whether I am right in that assumption. It must be remembered that these specimens were all hard-rolled to a very high tensile strength. Am I correct in assuming that a hard-rolled corrosion-resistant steel sheet will corrode somewhat more than one in the annealed condition and perhaps the fact that all these were hard-rolled to a high tensile strength may have some significance in the corrosion behavior shown by the author's illustrations.

MR. WALDRON.—I do not know whether I can comment on that, because these tests were made with thin high-strength corrosion-resistant steel sheets as used for aircraft purposes. As a result, they were in the cold-rolled condition to obtain a high strength. Samples in the annealed condition were not included.

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[1] Member of Firm, Singmaster and Breyer, New York, N. Y.

[2] Mr. Waldron presented the paper for Mr. Mutchler.—Ed.

[3] Metallurgist, National Bureau of Standards, Washington, D. C.

[4] In Charge of Corrosion Engineering Section, Development and Research Div., The International Nickel Co., Inc., New York, N. Y.

[5] Material and Process Standards Supervisor, Sperry Gyroscope Co., Inc., Great Neck, N. Y.

CHAIRMAN LAQUE.—If I may be permitted another observation other than as chairman, I can say I have tested cold-rolled 18-8 steel in sea water in the full-hard and in the annealed conditions and found no difference in susceptibility to corrosion of material having tensile properties within the limits we are talking about here. I believe there have been instances where wire has been so grossly cold-drawn as to affect its resistance to corrosion in some ways. I do not think the steel discussed here was influenced greatly in its corrosion by the fact it had been cold-rolled to the extent described.

MR. ASHKINAZY.—I was fully aware of the fact that the steels tested here were all in the hard-rolled condition and the fact that they were not annealed. I am glad to hear that there is practically no difference between the hard-rolled and annealed condition. However, if I remember correctly, there are some Government specifications which insist on the annealed condition of the 18-8 corrosion-resistant steel where a maximum corrosion resistance is required. I was just wondering why that was so.

CHAIRMAN LAQUE.—I am unable to suggest the basis of some of these specifications. I presume they are sometimes based on suspicion rather than fact.

MR. FRANK N. SPELLER.[6]—Does the author say anything about the influence of marine organisms?

MR. WALDRON.—There were no marine organisms on the tide water specimens at any time during their exposure.

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[6] Metallurgical Consultant, Pittsburgh, Pa.

ATMOSPHERIC CORROSION TESTS ON CORROSION-RESISTANT STEEL*

BY GRANT L. SNAIR, JR.[1]

The deterioration of metals exposed to the atmosphere is undoubtedly the most prevalent kind of corrosive attack. Although tests for the systematically organized study of atmospheric corrosion are time-consuming, they cannot be satisfactorily duplicated by the usual accelerated and carefully controlled laboratory tests which provide most of the data for metals exposed to the various corrosive media. Dependable data can be secured only after unsheltered metal specimens have been subjected to actual atmospheric conditions for many years without interruption in order that the total effect of the numerous factors contributing to atmospheric corrosion may be observed. Periodic inspections as the test progresses are also required in order properly to evaluate the results.

When considered from the corrosion standpoint, the effect of a particular atmosphere is dependent upon the presence of contaminating gases and suspended solid matter, as well as the moisture, or humidity and seasonal changes. The impurities may act to cause corrosion by direct chemical attack, or they may accumulate on the metal and invite or accelerate corrosion. When these deposits absorb water and any corrosive chemical agents present in the air, they are held in contact with the metal for long intervals and are less readily washed away by occasional rainfall.

Since weather conditions and the degree of pollution vary considerably throughout the year, and because corrosion-resisting metals are of primary interest in this study, long-range testing is necessary. Although the average corrosiveness of a certain atmosphere at a given location tends to be similar each year, it does not necessarily follow that the rate of attack or type of attack noted upon a particular specimen will be proportional to the number of years of exposure. Thus, the kind of oxide film formed upon the surface of the metal early in the exposure period may appreciably determine the extent of the corrosive attack for some time because the film may be of a protective nature.

This report describes the results of tests in which specimens of "stainless" steel alloys have been continually exposed to a severely corrosive industrial atmosphere for approximately ten years.

TEST METHOD

Location:

The exposure racks were installed on the property of the Allegheny Ludlum Steel Corp., Brackenridge, Pa., a heavy industrial area situated in a river valley about 20 miles from Pittsburgh. The atmosphere in this locality is contaminated with smoke, soot, sulfur dioxide, coal ash, lime, and iron dust. Passing locomotives also frequently introduce smoke and steam at the test location. The condensation of steam in a polluted atmosphere carries a greater number of suspended particles from the air to the specimens. When sulfur dioxide is present, dilute acid might be formed and deposited on the metal instead of being washed away, as by rainfall. In this climate, extreme weather conditions including fog, rain, and snow are encountered.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] Research Chemist, Allegheny-Ludlum Steel Corp., Brackenridge, Pa.

The corrosive conditions related to industrial atmospheres, however, are known to vary greatly at localized areas. For example, the pollution and condensation encountered at a locomotive roundhouse may cause severe corrosion of "stainless" steel within one year's time, while the same metal might remain practically unattacked at a location a hundred yards away. To further illustrate the effect of various industrial atmospheres, a type 410 corrosion-resistant steel roof at a glass plant two miles from Brackenridge has been in service for 20 years, and type 304 downspouts on homes in the community have been quite satisfactory. Accordingly, a location which was subject to an average industrial atmosphere was therefore selected for the tests.

Construction of the Rack:

The racks were constructed of A-shaped wooden end sections joined by horizontal wooden cross bars, from which the specimens were vertically supported by means of corrosion-resistant steel pegs. The cross bars were so arranged that any drippage from specimens on one cross bar could not fall upon the specimens located on lower cross bars, and the supporting pegs were spaced so that the specimens were well separated. The racks were built sufficiently high to prevent the splashing of the specimens with wet soil during heavy rains.

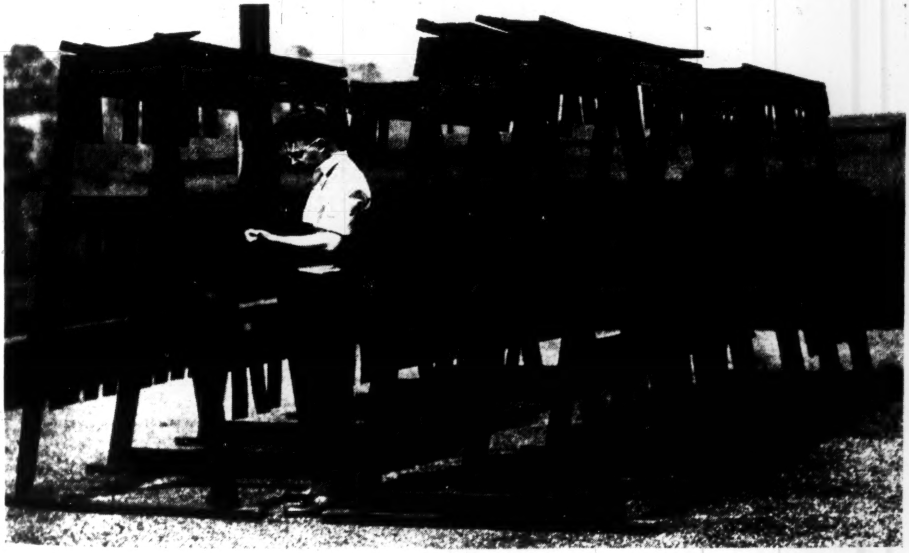

FIG. 1.—Exposure Rack for Atmospheric Corrosion Tests, Showing Construction and the Means by Which the Specimens Were Supported.

Figure 1 shows the construction of the exposure racks and the means by which specimens were supported for exposure. These particular racks are shown at a roof location.

Site:

The exposure racks were arranged on the ground at a test site which was located for eight years at a point in the plant yard where the pollution of the atmosphere was heaviest. Later, for the last two years, the racks were moved about a hundred yards east to the roof of a two story chemical laboratory building where fumes from the laboratory ducts were also encountered. Otherwise, the mill atmos pherewas identical. The racks were arranged without shelter in north-south positions so that the specimens faced east and west.

Specimen Size and Preparation:

The specimens were sheared uniform in size, 6 by 12 in. The effects of variations in corrosion-resistant steel alloy compositions, surface finishes, heat treatments, welding, and contact with other metals were studied in this project. Many of the specimens were prepared in duplicate and placed in adjacent positions on the exposure racks. All contamination which might have accumulated upon the metal during preparation and handling was removed by careful cleaning and degreasing immediately prior to exposure. A hole $\frac{3}{8}$ in. in diameter was drilled near one end of each specimen so that it could be suspended in a vertical position by means of a stainless type supporting rod. The upper 4 in. of the back of each specimen contacted the wooden cross bar, with the result that the area of contact was protected from direct exposure to the atmosphere.

Examination of the Specimens:

Each specimen was visually examined periodically when convenient in order to determine the progress of any corrosive attack. All specimens except those cleaned annually remained undisturbed upon the exposure rack during the entire 10-yr. testing period, and visual examinations were made with each specimen in its position on the rack. The specimens which were scheduled for yearly cleaning were examined before removal from the rack, after which they were carefully cleaned, inspected, and returned to their original places.

Accordingly, in cases where duplicate specimens were exposed, one was cleaned annually to permit a close inspection of the condition of the metal surface, while the companion specimen remained without cleaning during the entire testing period in order to keep the film of dirt and corrosion products in contact with the surface of the metal. At the conclusion of the test, the duplicates were cleaned for comparison.

In the early portion of the testing program, rather frequent examinations were made because slight changes in the appearance of the surfaces of some of the specimens were noticeable over periods of a few days or weeks. However, due to the corrosion-resisting properties of the alloys and because solid matter from the atmosphere accumulated to form an increasingly heavy layer on the metal surfaces, even semiannual and annual inspections in many instances revealed little or no change in the condition of the specimens. Consequently, the very frequent observations were of little importance, and the evaluation of the effects of weathering was based chiefly upon the annual notations and the final, very thorough inspections of the carefully cleaned surface of each specimen.

At the end of the testing period only half of the surface of each continuously exposed specimen was cleaned in order that a part of the layer of dirt and corrosion products might be retained for further testing. In general, the right half of each specimen was cleaned, but in the case of welded specimens in which the weld deposits extended from end to end through the centers of the specimens, the upper halves were cleaned to permit examination of both sides of the welds.

TABLE I.—THE STRAIGHT CHROMIUM CORROSION-RESISTANT STEEL ALLOYS.

Analysis	Heat Treatment	Surface Finish
12 PER CENT CHROMIUM, TWELVE SPECIMENS TESTED, COMPOSITION RANGE 11.50 TO 13.50 PER CENT CHROMIUM		
1. 0.088 C—13.11 Cr—0.338 Ni	Fully annealed	Mirror buffed, cleaned yearly
2. Same heat (duplicate specimen)	Fully annealed	Mirror buffed
3. Same heat	Fully annealed	Regular polished, cleaned yearly
4. Same heat (duplicate specimen)	Fully annealed	Regular polished
5. Same heat	Fully annealed	Rough polished, cleaned yearly
6. Same heat (duplicate specimen)	Fully annealed	Rough polished
7. Same heat	Fully annealed	Sandblasted and dipped in 10% HNO_3, 160 F., cleaned yearly
8. Same heat (duplicate specimen)	Fully annealed	Sandblasted and dipped in 10% HNO_3, 160 F.
9. Same heat	Fully annealed	Sandblasted and pickled
10. Same heat	Fully annealed	Sandblasted only
11. Same heat	Fully annealed; electric welded	Sandblasted
12. Same heat	Fully annealed; electric welded	Weld ground flush and entire surface polished
14 PER CENT CHROMIUM, TEN SPECIMENS TESTED		
1. 0.074 C—14.46 Cr—0.251 Ni	Fully annealed	Mirror buffed, cleaned yearly
2. Same heat (duplicate specimen)	Fully annealed	Mirror buffed
3. Same heat	Fully annealed	Regular polished, cleaned yearly
4. Same heat (duplicate specimen)	Fully annealed	Regular polished
5. Same heat	Fully annealed	Rough polished, cleaned yearly
6. Same heat (duplicate specimen)	Fully annealed	Rough polished
7. Same heat	Fully annealed	Sandblasted and dipped in 10% HNO_3 160 F., cleaned yearly
8. Same heat (duplicate specimen)	Fully annealed	Sandblasted and dipped in 10% HNO_3, 160 F.
9. Same heat	Fully annealed	Sandblasted and pickled
10. Same heat	Fully annealed	Sandblasted
17 PER CENT CHROMIUM, TWELVE SPECIMENS TESTED, COMPOSITION RANGE 16 to 18 PER CENT CHROMIUM		
1. 0.11 C—16.80 Cr—0.354 Ni	Fully annealed	Mirror buffed, cleaned yearly
2. Same heat (duplicate specimen)	Fully annealed	Mirror buffed
3. Same heat	Fully annealed	Regular polished, cleaned yearly
4. Same heat (duplicate specimen)	Fully annealed	Regular polished
5. Same heat	Fully annealed	Rough ground, cleaned yearly
6. Same heat (duplicate specimen)	Fully annealed	Rough ground
7. Same heat	Fully annealed	Sandblasted and dipped in 10% HNO_3, 160 F., cleaned yearly
8. Same heat (duplicate specimen)	Fully annealed	Sandblasted and dipped in 10% HNO_3, 160 F.
9. Same heat	Fully annealed	Sandblasted and pickled
10. Same heat	Fully annealed	Sandblasted only
11. Same heat	Fully annealed; electric welded	Sandblasted
12. Same heat	Fully annealed; electric welded	Weld ground flush and entire surface polished
22.5 PER CENT CHROMIUM, TEN SPECIMENS TESTED		
1. 0.095 C—22.51 Cr—0.813 Ni	Fully annealed	Mirror buffed, cleaned yearly
2. Same heat (duplicate specimen)	Fully annealed	Mirror buffed
3. Same heat	Fully annealed	Regular polished, cleaned yearly
4. Same heat (duplicate specimen)	Fully annealed	Regular polished
5. Same heat	Fully annealed	Rough ground, cleaned yearly
6. Same heat (duplicate specimen)	Fully annealed	Rough ground
7. Same heat	Fully annealed	Sandblasted and dipped in 10% HNO_3, 160 F., cleaned yearly
8. Same heat (duplicate specimen)	Fully annealed	Sandblasted and dipped in 10% HNO_3, 160 F.
9. Same heat	Fully annealed	Sandblasted and pickled
10. Same heat	Fully annealed	Sandblasted only
28 PER CENT CHROMIUM, TWELVE SPECIMENS TESTED, COMPOSITION RANGE 23 TO 30 PER CENT CHROMIUM		
1. 0.17 C—27.39 Cr—0.495 Ni	Fully annealed	Mirror buffed, cleaned yearly
2. Same heat (duplicate specimen	Fully annealed	Mirror buffed
3. Same heat	Fully annealed	Regular polished, cleaned yearly
4. Same heat (duplicate specimen)	Fully annealed	Regular polished
5. Same heat	Fully annealed	Rough ground, cleaned yearly
6. Same heat (duplicate specimen)	Fully annealed	Rough ground
7. Same heat	Fully annealed	Sandblasted and dipped in 10% HNO_3, 160 F., cleaned yearly
8. Same heat (duplicate specimen)	Fully annealed	Sandblasted and dipped in 10% HNO_3, 160 F.
9. Same heat	Fully annealed	Sandblasted and pickled
10. Same heat	Fully annealed	Sandblasted only
11. Same heat	Fully annealed; electric welded	Sandblasted
12. Same heat	Fully annealed; electric welded	Weld ground flush and entire surface polished

TABLE II.—THE UNMODIFIED CHROMIUM-NICKEL CORROSION-RESISTANT STEEL ALLOYS.

Analysis	Heat Treatment	Surface Finish
TYPE 301, ONE SPECIMEN TESTED, COMPOSITION RANGE 0.09 TO 0.20 PER CENT C—16 TO 18 PER CENT Cr—6 TO 8 PER CENT Ni		
1. 0.15 C 1.13 Mn 16.17 Cr 6.95 Ni	Fully Annealed	White pickled
TYPE 302, TWENTY-FIVE SPECIMENS TESTED, COMPOSITION RANGE 0.08 MINIMUM TO 0.20 PER CENT C—17 TO 19 PER CENT Cr—8 TO 10 PER CENT Ni		
1. 0.088 C 19.18 Cr 8.80 Ni	Fully annealed	Mirror buffed, cleaned yearly
2. Same heat (duplicate specimen)	Fully annealed	Mirror buffed
3. Same heat	Fully annealed	Regular polished, cleaned yearly
4. Same heat (duplicate specimen)	Fully annealed	Regular polished
5. Same heat	Fully annealed	Tampico brushed, cleaned yearly
6. Same heat (duplicate specimen)	Fully annealed	Tampico brushed
7. Same heat	Fully annealed	Rough ground, cleaned yearly
8. Same heat (duplicate specimen)	Fully annealed	Rough ground
9. 0.10 C 19.00 Cr 9.11 Ni	Fully annealed	Regular polished
10. 0.11 C 19.41 Cr 9.02 Ni	Fully annealed	White pickled
11. Same heat	Fully annealed + 1200 F., 1 hr.	White pickled
12. Same heat	Fully annealed base metal; electric welded	Welding flux and scale removed by pickling
13. Same heat	Fully annealed base metal; electric welded	Weld deposit ground flush with the base metal (Grade "A" welding rod used)
14. 0.17 C 17.66 Cr 8.19 Ni	Fully annealed	White pickled
15. Same heat	Fully annealed + 1200 F., 1 hr.	White pickled
16. Same heat	Fully annealed base metal; electric welded	Weld deposit ground flush with the base metal
17. 0.11 C 19.69 Cr 9.19 Ni	Fully annealed	White pickled
18. Same heat	Fully annealed	Regular polished
19. 0.11 C 19.20 Cr 8.44 Ni	Fully annealed	White pickled
20. 0.084 C 18.61 Cr 7.79 Ni	Fully annealed	White pickled
21. 0 10 C 16.90 Cr 9.63 Ni Strip	Fully annealed	Cold-rolled
22. 0.10 C 18.16 Cr 8.60 Ni Strip	Fully annealed	Cold-rolled
23. 0.11 C 17.92 Cr 8.42 Ni Strip	Fully annealed	Cold-rolled
24. 0.10 C 18.97 Cr 8.69 Ni Strip	Fully annealed	Cold-rolled
25. 0.096 C 18.65 Cr 8.86 Ni	Hot-rolled	White pickled
TYPE 304, ELEVEN SPECIMENS TESTED, COMPOSITION RANGE 0.08 PER CENT C MAXIMUM—18 TO 20 PER CENT Cr—8 TO 10 PER CENT Ni		
1. 0.067 C—18.92 Cr—8.88 Ni	Fully annealed	White pickled
2. Same heat (duplicate specimen)	Fully annealed	White pickled
3. Same heat	Fully annealed	White pickled + regular five pass cold-rolled smoothing finish
4. Same heat	Fully annealed	Sandblasted only
5. Same heat	Fully annealed	Sandblasted + 10% HNO_3 dip
6. Same heat	Fully annealed	Sandblasted and pickled
7. Same heat	Fully annealed	White pickled and passivated in solution containing 4% chromic acid and 4% HF
8. Same heat	Fully annealed + 1200 F., 1 hr.	White pickled
9. Same heat	Fully annealed base metal; electric welded	Grade A welding rod used; white pickled
10. Same heat	Fully annealed base metal; electric welded	Grade A welding rod used; weld deposit ground flush with the base metal
11. Same heat	Fully annealed base metal; electric welded	Grade A welding rod used; weld deposit ground flush with the base metal and entire surface regular polished
TYPE 308, FOUR SPECIMENS TESTED, COMPOSITION RANGE 0.08 PER CENT C MAXIMUM—19 TO 21 PER CENT Cr—10 TO 12 PER CENT Ni		
1. 0.062 C—20.65 Cr—9.98 Ni	Fully annealed	White pickled
2. Same heat (duplicate specimen)	Fully annealed	White pickled
3. Same heat	Fully annealed	Regular polished
4. Same heat	Fully annealed + 1200 F., 1 hr.	Sandblasted and white pickled

TABLE II.—*Continued.*

Analysis	Heat Treatment	Surface Finish
TYPE 309, TWO SPECIMENS TESTED, COMPOSITION RANGE 0.20 PER CENT C MAXIMUM—22 TO 24 PER CENT Cr—12 TO 15 PER CENT Ni		
1. 0.18 C—22.65 Cr—11.57 Ni	Fully annealed	White pickled
2. Same heat	Fully annealed + 1200 F., 1 hr.	White pickled
24 PER CENT CHROMIUM—16 PER CENT NICKEL, ONE SPECIMEN TESTED		
1. 0.11 C—24.20 Cr—16.40 Ni	Fully annealed	White pickled

Description of Test Results:

The rates of corrosive attack upon the specimens tested are not reported in terms of weight losses. The weighing of such large specimens is impractical, and because the atmospheric corrosion of corrosion-resistant steel is of a localized nature, any weight losses so obtained would not be significant. No data from tension tests or measurements of the depth of pitting can be offered at this time. It was also impossible to show suitably by means of photographs the true appearance of the metal surfaces. The information presented in this report is based solely upon the visual examination of the front surfaces of the specimens during a 10-yr. testing period of exposure to the atmosphere and at the conclusion of the test. All specimens were examined under a 15-power binocular microscope.

The corrosion of the specimen is mentioned for the most part in terms of pitting or rusting. As applied in this case, the term "pitting" should not be associated with large, deep cavities, but only to exceptionally fine, shallow surface flaws which in no case destroyed the usefulness of the metal. When rusting occurred, it usually appeared in the form of either very light spots, streaks, stains, or films.

The resistance of the stainless type steel alloys to atmospheric corrosion

TABLE III.—THE MODIFIED CHROMIUM-NICKEL CORROSION-RESISTANT STEEL ALLOYS.

Analysis	Heat Treatment	Surface Finish
TYPE 347, TWO SPECIMENS TESTED, COMPOSITION RANGE 0.10 PER CENT C MAXIMUM—17 TO 19 PER CENT Cr—9 TO 12 PER CENT Ni—8 × PER CENT C MINIMUM Cb		
1. 0.080 C—19.93 Cr—8.97 Ni—0.82 Cb	Fully annealed	White pickled
2. Same heat	Fully annealed + 1200 F., 1 hr.	White pickled
TYPE 321, FIVE SPECIMENS TESTED, COMPOSITION RANGE 0.10 PER CENT C MAXIMUM—17 TO 19 PER CENT Cr—8 TO 11 PER CENT Ni—4 × PER CENT C MINIMUM Ti		
1. 0.063 C—17.10 Cr—8.96 Ni—0.36 Ti	Fully annealed	White pickled
2. Same heat	Fully annealed + 1200 F., 1 hr.	White pickled
3. 0.11 C—17.38 Cr—8.99 Ni—0.34 Ti	Fully annealed	White pickled
4. Same heat	Fully annealed + 1200 F., 1 hr.	White pickled
5. Same heat	Fully annealed base metal; electric welded	Sandblasted
TYPE 316, TWO SPECIMENS TESTED, COMPOSITION RANGE 0.10 PER CENT C MAXIMUM—16 TO 18 PER CENT Cr—10 TO 14 PER CENT Ni—2 TO 3 PER CENT Mo		
1. 0.086 C—19.27 Cr—9.29 Ni—2.54 Mo	Fully annealed	White pickled
2. Same heat	Fully annealed + 1200 F., 1 hr.	White pickled
18-8 PLUS VANADIUM, TWO SPECIMENS TESTED		
1. 0.066 C—18.86 Cr—9.15 Ni—1.29 V	Fully annealed	White pickled
2. Same heat	Fully annealed + 1200 F., 1 hr.	White pickled

may be compared with that offered by specimens of unpainted common steel, 0.050 in. in thickness, which were exposed under identical conditions. After 4 yr., the thickness of the common steel specimens had been reduced approximately one half by rusting, and complete deterioration occurred within eight to nine years.

MATERIALS TESTED

Tables I to III furnish complete information concerning the composition, heat treatment, surface finish, and welding of the corrosion-resistant steel alloys tested.

CORROSION-RESISTANT STEEL TYPE 304 IN CONTACT WITH OTHER METALS

Specimens were prepared to determine whether galvanic effects would be induced by the contact of corrosion-resistant steel with other metals. The upper or lower half of each specimen consisted of white pickled, cold-rolled, smoothed corrosion-resistant steel type 304 material to which another metal was riveted to complete the specimen. The upper half overlapped the lower half by 1 in., and the pieces were tightly joined with three type 304 rivets.

The specimens were examined without cleaning during the test. After 10-yr. exposure, they were carefully cleaned to determine the condition of the metal surface.

The tests were conducted only to study the effects of such contact on the corrosion resistance of the stainless type steel. The corroded surfaces of the other metals were not considered.

The following are the materials which were riveted to type 304 steel white pickled and regular 5 pass cold-rolled smooth-finished sheet, composition 0.067 per cent carbon, 18.92 per cent chromium, and 8.88 per cent nickel:

1. Pure aluminum, bright cold-rolled
2. Pure zinc, bright cold-rolled
3. Pure lead, pickled in 10 per cent HCl
4. Pure copper, bright cold-rolled
5. Pure nickel, white pickled
6. Monel metal, cold-rolled
7. Pure tin, cold-rolled
8. Common steel, pickled

SUMMARY OF RESULTS

The information derived from the study of the individual groups and a comparison of all the groups provides a basis for a number of pertinent general conclusions which may be briefly summarized, while a general rating of the steels is shown in Table IV.

1. When several specimens from a particular heat with identical surface finishes were tested, excellent correspondence of results was noted in a good majority of cases. Consequently, although some of the chromium-nickel alloys were represented by only a few specimens, it would appear that the results could be given equal weight with the groups including larger numbers of specimens.

2. All of the corrosion-resistant steel alloys in the fully annealed condition offer excellent, but not complete, resistance to atmospheric corrosion. Most of them are subject to slight attack by pitting and by rusting.

3. The addition of chromium to steel increases its resistance to atmospheric corrosion. The straight chromium corrosion-resistant steel alloys, containing from 12 to 28 per cent chromium, showed that the corrosion resistance increases as the chromium content is increased. For example, the lowest alloy group tested, which contained 12 per cent chromium, was attacked by general

fairly heavy rusting and fine pitting profusely distributed over the surfaces. After final cleaning, the effects of corrosion were plainly visible upon all specimens. The higher chromium alloys of this series showed lighter rusting and less pitting. Accordingly, the 28 per cent chromium group, the highest straight chromium alloy tested, was subject to light rusting and exceptionally slight, scattered pitting. The pitting on the cleaned surfaces could be observed only upon close inspection and, in some cases, by examination under a binocular microscope.

TABLE IV.—CLASSIFICATION OF RESISTANCE TO ATMOSPHERIC CORROSION.

In the following table the alloy compositions are listed in eight general classes of resistance to atmospheric corrosion. The rating is based upon the final appearance of fully annealed material exposed continuously for ten years. The best alloys are arranged at the top of the list.

Rating	Alloy Grade	Most Resistant Finishes	
I	316	1. Only the white pickled finish was tested	
II	28 per cent Cr	1. Mirror buffed	2. Regular No. 4 polished
III	301	1. Only the white pickled finish was tested	
	302	1. Mirror buffed	2. Regular No. 4 polished
	304	1. White pickled and passivated (only white pickled finishes and sandblasted finishes were tested)	2. White pickled + regular 5 pass cold-rolled smoothed finish
	308	1. Regular No. 4 polished	2. White pickled
	309	1. Only the white pickled finish was tested	
	24 per cent Cr—16 per cent Ni	1. Only the white pickled finish was tested	
	347	1. Only the white pickled finish was tested	
	18-8 plus vanadium	1. Only the white pickled finish was tested	
IV	321	1. Only the white pickled finish was tested	
V	22.5 per cent Cr	1. All of the sandblasted finishes	2. Mirror buffed
VI	17 per cent Cr	1. Sandblasted + 10 per cent HNO_3 dip, 160 F.	2. Sandblasted and pickled
VII	14 per cent Cr	1. Sandblasted only	2. Sandblasted and dipped in 10 per cent HNO_3, 160 F.
VIII	12 per cent Cr	1. Mirror buffed	2. Regular polished

The testing of similarly finished specimens of each composition in the fully annealed condition permitted accurate conclusions based upon alloy content.

4. The corrosion-resistant steel alloys containing chromium and nickel also resist atmospheric corrosion. Certain compositions are slightly more resistant than others. In the fully annealed condition, the carbon content was unimportant within the range covered by the specimens tested (0.05 to 0.20 per cent). Types 301, 302, and 304 offered better resistance to pitting and rusting than all of the members of the straight chromium series except the 28 per cent chromium group.

These three grades of chromium-nickel corrosion-resistant steel showed only very light rust formations and localized, widely separated areas of very fine pitting in the form of minute, shallow, white specks which barely interrupted the surface of the metal. Essentially, the original appearance of the specimens remained unchanged by the corrosive attack.

Any increase in the chromium and nickel content above 18 per cent chromium and 8 per cent nickel did not appreciably improve the resistance to rusting and pitting. The type 308 group (20 per cent chromium, 10 per cent nickel) were subject to very slight rusting and pitting. While most of the pits were very fine and shallow, many of them were rather broad. The type 309 specimen (23 per cent chromium, 12 per cent nickel) showed a few small, lightly rusted areas and very fine general and localized pitting. Even the 24 per cent chromium, 16 per cent nickel specimen was attacked with very fine, shallow, general pitting well distributed over the entire surface, but its appearance was practically unaffected. Thus, some of the high alloy specimens showed somewhat greater corrosion than the types 301, 302, and 304 specimens.

5. The corrosion resistance of the stainless type steel alloys can be altered to some extent by the application of various surface finishes. Where surface finishes were compared after testing, this was the only variable. Many of the chromium-nickel steel specimens were tested only in the white pickled condition. When polished surfaces were included, the mirror buffed, regular No. 4 polished, and the Tampico brushed or white pickled finishes seemed most effective in the order listed. For all of the straight chromium groups except 12 per cent chromium, the sandblasted surfaces resisted rusting and pitting better than any of the polished surfaces. The mirror buffed and the regular No. 4 polished 12 per cent chromium specimens were best. Inasmuch as none of the specimens tested was severely pitted or rusted, the effect of different finishes might, for all general purposes, be considered relatively unimportant.

6. The heat treatment of the unmodified chromium-nickel corrosion-resistant steel alloys at 1200 F., the temperature range in which carbide precipitation occurs, only slightly decreases the resistance of the metal to atmospheric corrosion. As an interesting comparison, these metals failed after exposure to boiling Krupp solution (10 per cent copper sulfate and 10 per cent sulfuric acid) for periods ranging from 8 to 26 hr. The specimens were subject to a little more rusting and general and localized pitting than the fully annealed material. The specimens containing the greater percentages of carbon seemed more susceptible to pitting and intergranular attack. All of the specimens tested revealed slight indications of intergranular attack. Some of the corroded areas were darker than the original surfaces, but no severe attack was encountered in any case.

7. The modification of the chromium-nickel corrosion-resistant steel alloys with either columbium, titanium, or vanadium does not improve the corrosion resistance of the metal in the fully annealed condition. All of these modifications considerably improved the resistance of the metal which was heat treated at 1200 F. Except for the type 321 containing titanium which was less resistant to rusting than any of the others, each of these heat-treated modified alloys offered equal general resistance to rusting and to the very fine localized and general pitting. No intergranular attack occurred. The specimens compared favorably with the fully annealed unmodified alloys.

8. The modification of the chromium-nickel corrosion-resistant steel alloys with 2 to 3 per cent molybdenum improves the resistance of the fully annealed metal to pitting. It also seems to improve the corrosion resistance of the material heat-treated at 1200 F.

When the fully annealed white pickled specimen was cleaned after 10-yr. exposure, the surface showed no evidence of corrosive attack by pitting. The heat-treated, white pickled specimen remained unattacked except for a few exceedingly fine shallow pits.

9. The hot-rolled type 302 specimen closely resembled specimens of type 302 which had been heat-treated at 1200 F. for 1 hr. The similarity was probably due to the presence of precipitated carbides in both materials.

10. The cold-rolled type 302 strip finishes produced varying results. Although the specimens represented different heats, the compositions did not vary greatly. The corrosion resistance of cold-rolled type 302 strip compares favorably with that of the other smoothly

finished type 302 specimens in sheet form.

11. The undisturbed specimens offered greater resistance to pitting. When duplicate specimens having identical composition, heat treatment, and surface finish were exposed, a slightly greater degree of attack, both in size and number of the pits, was generally noted on the annually cleaned specimen.

12. The welding of the stainless type steel alloys causes little or no decrease in their resistance to atmospheric corrosion. In most cases, the welds which were ground flush with the base metal and smoothly polished offered better corrosion resistance than the unfinished or roughly ground welds. The tests indicate that when unmodified chromium-nickel corrosion-resistant steel is welded, the areas of the base metal which are immediately adjacent to the weld and subject to heating effects are slightly more susceptible to rusting, pitting, and intergranular attack. The welded straight-chromium specimens which were tested showed no decrease in the corrosion resistance of the weld deposit or the areas of the base metal adjacent to the weld.

13. Passivation with either 10 per cent nitric acid or 4 per cent chromic acid and 4 per cent hydrofluoric acid solutions did not noticeably improve the corrosion resistance of the specimens.

14. No severe galvanic action occurred on any of the riveted specimens which were tested. In most cases, more attack was observed along the edges and the end away from the junction of the two metals. This corrosion compared closely with the very fine pitting encountered along the edges of the nonriveted type 304 specimens. The type 304 upper halves of the specimens showed less attack by rusting and pitting than the type 304 lower halves which were subject to the heavier accumulation of dirt and the corrosion products washed from the other metal by rain. It was noted that the lower ends of all of the nonriveted specimens were also corroded more than the rest of the surfaces. It is therefore unlikely that any of the corrosion of these specimens was caused by galvanic action.

WEATHERING BEHAVIOR OF CORROSION-RESISTANT STEEL INSECT SCREENS*

By W. A. Wesley[1] and H. R. Copson[1]

Synopsis

The results of several series of atmospheric corrosion tests are presented in which commercially woven wire-cloth screens of bronze, 18 per cent chromium, 8 per cent nickel corrosion-resistant steel and type 316 corrosion-resistant steel were exposed in sulfurous, industrial, marine, and rural atmospheres. It is brought out that the atmospheric corrosion of wire cloth is influenced to a greater extent than that of perhaps any other useful form of a metal by the manner in which the cloth is exposed, or more exactly, by the degree of shelter from the rinsing action of rain.

Although some of the tests will not mature for years, certain conclusions can be drawn at this stage. It is evident that 18-8 corrosion-resistant steel wire cloth is too susceptible to pitting if partly sheltered from rain in industrial and marine atmospheres to outlast bronze similarly sheltered. Type 316 corrosion-resistant steel screens are showing phenomenal resistance to corrosion in industrial atmospheres whether sheltered or not and will long outlive bronze screens. A word of caution is necessary, however, since a few type 316 screens which contained some 18 per cent chromium, 8 per cent nickel corrosion-resistant steel wires by mistake, were rapidly pitted where rust from the latter contacted the former. In marine atmospheres the type 316 screens develop faint rust spots and some pitting but will show satisfactory life. The molybdenum content should be high.

Other advantages of type 316 corrosion-resistant steel insect screens over bronze screens include freedom from staining of white paint, high strength, clearer vision, and greater ease of cleaning.

In contrast with the copious literature on the corrosion resistance of other useful forms of metal there is little information available on the atmospheric durability of insect screen-wire cloth. A 9-yr. test of non-ferrous wire cloths was carried out by the National Bureau of Standards[2] in cooperation with the former A.S.T.M. Committee D-14 on Screen-Wire Cloth[3] beginning in 1925. Seven materials were exposed at each of four test sites in a vertical position with no shelter and no backing, so that there was free access of wind and rain to all parts of the specimens except the very edge. None of the materials displayed noteworthy improvement over the so-called "bronze" insect screen of 90 per cent copper, 10 per cent zinc (commercial bronze) which is accepted as the standard commercial product in the United States.

It is generally agreed that any new insect screen material to be commercially successful in the United States must either provide superior corrosion resistance to or be less costly than bronze. An important disadvantage of bronze screens is that their corrosion products tend to stain white paints and masonry over which rainwater may run as it drains from the screen.

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[1] Assistant Director and Research Chemist, respectively, Research Laboratory, The International Nickel Co. Inc , Bayonne, N. J.

[2] G. W. Quick, "Atmospheric Exposure Tests on Non-Ferrous Screen Wire Cloth," *Journal of Research*, Nat. Bureau of Standards, Vol. 14, p. 775 (1935).

[3] Discontinued in 1937.

This laboratory became interested as early as 1932 in the possibility that stainless steel screens might combine adequate corrosion resistance with freedom from paint staining. Previous experience had proven repeatedly that the longevity and weathering behavior of woven cloth cannot be safely predicted from the results of exposure of unwoven wire or of sheet metal nor even from screen exposure tests of short duration. The physical structure of screen cloth, with its multiplicity of contacts and the effect of wire intersections in retaining and stabilizing droplets of liquid, make it uniquely susceptible to the detrimental effect of shelter from rain. If areas of the screen are shielded from the direct rinsing action of rain, as is true when screens are installed in porch windows or doors, dirt and corrosion products accumulate on the screen. These may become wet on humid days by deliquescence, while boldly exposed, well washed screens remain dry.

TABLE I.—OUTLINE OF EXPOSURE TESTS.

Test	Screen	Location	Shelter
Box exposures (exposed in 1932)	18-8 steel	Pittsburgh, Pa. North Brigantine, N. J.	Boldly exposed Boldly exposed
1932 tests	Bronze 18-8 steel	Bayonne, N. J. Basking Ridge, N. J. Jersey City, N. J. Fire Island, N. Y.	Partly sheltered Partly sheltered Partly sheltered Partly sheltered
1933 tests	Bronze 18-8 steel	Bayonne, N. J.	Completely sheltered and Boldly exposed
1935 tests	Type 302 steel Type 316 steel	North Brigantine, N. J.	Partly sheltered
1937 tests	Bronze Type 316 steel	Bayonne, N. J.	Partly sheltered Completely sheltered Boldly exposed
		Basking Ridge, N. J. Jersey City, N. J. Fire Island, N. Y.	Partly sheltered Partly sheltered Partly sheltered
1941 tests	Bronze Type 316 steel	Block Island, R. I.	Completely sheltered and Boldly exposed
1943 tests	Bronze Type 316 steel	Bayonne, N. J.	Completely sheltered and Boldly exposed
		Jersey City, N. J.	Partly sheltered
		Block Island, R. I.	Completely sheltered and Boldly exposed

OUTLINE OF EXPOSURE TESTS

In 1932 and 1933 three sets of atmospheric exposure tests were started to evaluate the merit of 18-8 corrosion-resistant steel as insect screen. In two of these tests a direct comparison was made with bronze insect screen. It soon became apparent that 18-8 steel would not be suitable under all conditions of exposure. In 1935, at the suggestion of R. J. McKay,[4] a type 316 molybdenum corrosion-resistant steel screen was exposed to a marine atmosphere. This screen seemed to be standing up so well that in 1937 more comprehensive exposures were started at four locations, and bronze screen included. The marine exposure of the 1937 tests was lost in the hurricane of 1938, and replaced by a new marine test in 1941. In 1943 additional tests were started comparing a third lot of type 316 screen with bronze. For complete outline of exposure tests, see Table I.

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[4] Private communication from R. J. McKay, International Nickel Co., Inc., New York, N. Y.

TABLE II.—SCREEN MATERIAL.

Material	Test	Screen Mesh	Wire Diameter, in.	Composition, per cent						Average Initial Breaking Load of Individual Wires, lb.	
				Copper	Zinc	Carbon	Nickel	Chromium	Molybdenum	Warp	Shoot
Bronze	1932	16	0.011	90.50	9.39						8.1
	1933	16	0.011	90.50	9.39					7.3	8.0
	1937	16	0.011	90.50	9.39					7.21[a]	
	1941	16	0.011	90.50	9.39					7.21[a]	
	1943	16	0.011	90.50	9.39					7.21[a]	
18-8 steel	Box exposures	16	0.010								9.1
	1932	16	0.010			0.13	8.34	18.34			9.7
	1933	16	0.010							9.5	9.2
	1935	16									
Type 316 molybdenum steel	1935	16				0.07	7.88	18.0	2.81	7.5	
	1937 and 1941	16	0.009			0.15	7.82	17.70	3.15[c]	7.38[b]	
	1943	16	0.009			0.07	12.44	18.27	2.13	6.5	

[a] Average of 108 wires, maximum 7.6 lb., minimum 6.8 lb., standard deviation 0.155 lb.
[b] Average of 120 wires, maximum 7.8 lb., minimum 6.9 lb., standard deviation 0.149 lb.
[c] 2.95 per cent in a separate analysis.

SCREEN MATERIALS

All samples of bronze screen were cut from one roll which was purchased in the open market. The bronze analyzed 90.50 per cent copper, 9.39 per cent zinc, and contained no tin. The screen was 16 mesh and the wires were 0.011 in. in diameter. The shoot wires were a little harder than the warp wires, although the wires in both directions of weaving were relatively hard.

All samples of 18-8 corrosion-resistant steel screen were probably from one lot, with the possible exception of the 1935 tests. An analysis of the screen used in the 1932 tests showed 0.13 per cent carbon, 8.34 per cent nickel, and 18.34 per cent chromium. The 18-8 corrosion-resistant steel screen was 16 mesh and had 0.010-in. diameter wires.

Three lots of type 316 molybdenum corrosion-resistant steel screen were represented in the tests. The analyses are in Table II. They were 16 mesh and had 0.009-in. wires. As described under the 1943 tests the third lot was found to have a few regular 18-8 warp wires woven into the screen. These wires were removed before the analyses were made.

TEST SITES

The locations in Table I represent four types of atmospheres: industrial, rural, sulfurous, and marine. Bayonne, N. J., and Pittsburgh, Pa., were industrial locations. At Bayonne the screens were in the laboratory yard, not far from a center of oil-refining activity. At Pittsburgh the screens were on the roof of a building adjacent to one of the railroad yards, a smoky spot. Basking Ridge, N. J., was a rural location; the screens were in a pasture on a farm 7 miles from the nearest large town. Jersey City, N. J., was a sulfurous atmosphere; the screens were on the roof of a passenger train shed where there was plenty of highly sulfurous smoke from coal-burning locomotives.

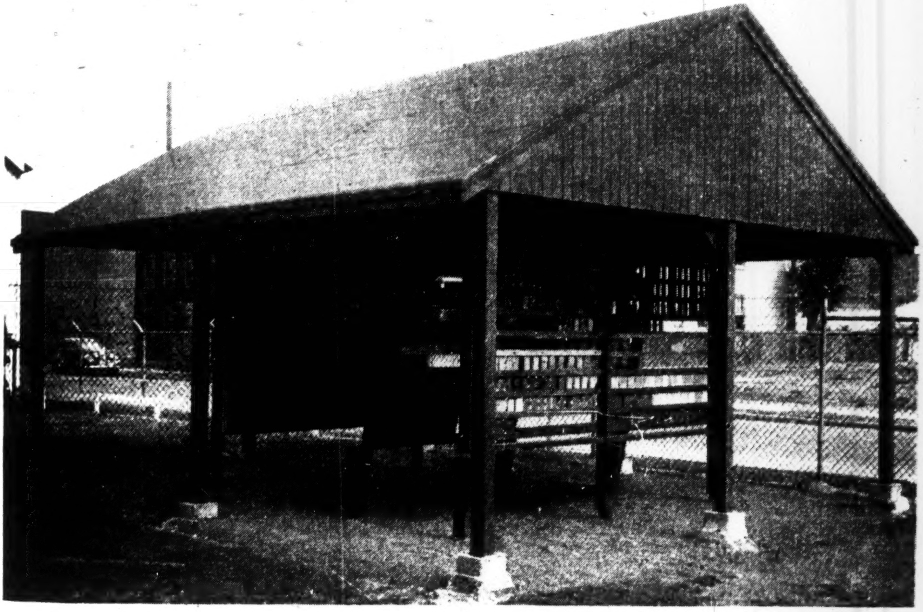

FIG. 1.—Exposure Rack at Bayonne Providing Complete Shelter.

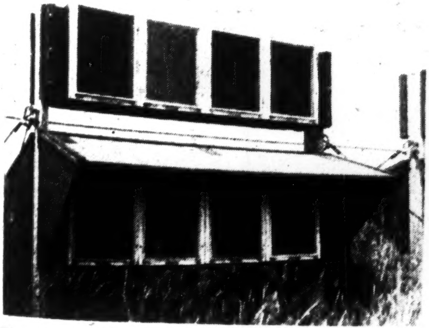

Fig. 2.—Exposure Racks at Block Island Providing Bold Exposure and Complete Shelter.

The remaining locations were marine. At North Brigantine, N. J., the screens were close to the shore. At Fire Island, N. Y., they were about 200 yd. from the ocean beach. At Block Island, R.I., the screens were on top of a 110 to 120-ft. bluff overlooking the ocean. All the marine locations were remote from manufacturing centers.

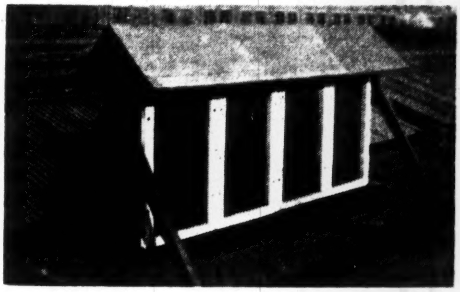

FIG. 3.—Exposure Rack at Jersey City Providing Partial Shelter.

EXPOSURE RACKS

Four types of racks were used which provided three kinds of shelter: complete shelter, partial shelter, and no shelter (bold exposure). The sheltered rack at

Bayonne is shown in Fig. 1 at the far left. The shelter was provided by a roofed shed open on all sides with an angle of 45 deg. between the lower edge of the eaves and the lowest point on the lowest specimen. Thus rain would have to descend at an angle greater than 45 deg. from the vertical before washing could occur on any screen. Drainage troughs were placed between each row of screens to prevent drip from any screen running down onto the one below. At Bayonne the boldly exposed rack was similar except that it was placed in the open without any shelter.

The type of rack used at Block Island is shown in Fig. 2. The idea was to provide complete shelter and bold exposure as at Bayonne, but on a smaller scale. The roof over the protected portion could be raised to permit easy installation and inspection of the screens.

All the racks providing partial shelter were similar to the Jersey City rack shown in Fig. 3. The top portion of the screens was tucked under a projecting eave such that about the upper third of the screens, while open to the air, was protected from the wash of rain. In testing screen exposed on these racks the upper partly sheltered portion was frequently compared with the lower unsheltered portion.

The fourth type of rack was used only in the box exposures. It consisted simply of a box with the test samples mounted on the sides and top. No shelter was provided.

The method of mounting the screens on the racks was the same in all tests. Small pieces of screen, varying in size from 7 by 18 in. to 18 by 34 in., were tacked to white painted wooden frames, with copper tacks in the case of bronze screens, and monel or Inconel tacks in corrosion-resistant steel. Half-round $\frac{1}{2}$-in. wood molding was fastened over the edge of the screen with Inconel brads. The screen frames were then mounted on the solid wall of the rack in such fashion as to form a box-like space about 3 in. deep behind each screen. This permitted air to circulate but prevented wind from blowing through. Screens could be mounted on both sides of the racks. The racks were painted dark green to facilitate visual examination of the screen.

METHODS OF MEASURING EXTENT OF CORROSION

Periodically the screens were inspected carefully while still on racks. At less frequent intervals strips of screen were cut out and brought to the laboratory.

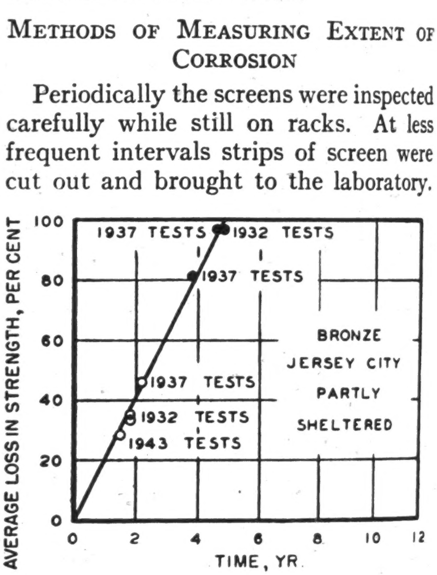

Fig. 4.—Consistency of Results.
Blacked-in points indicate some wire failures.

Individual wires were removed, their breaking strength determined and compared with the breaking strength of uncorroded wires. In most cases tension tests were made on at least 10 corroded wires. The remainder of the strip was then examined under a low-powered microscope both as-received and with the corrosion product removed.

It developed that, in general, bronze screen corroded more or less uniformly, while if corrosion occurred on the corrosion-resistant steel screens it was in the form of local pits. The tension tests were a sensitive means of detecting such pitting. Where the pitting was extremely local there was wide scatter in the breaking strengths of the wires. Only the average loss in breaking strength is recorded in the tables and graphs, and in addition complete breaks caused by corrosion of the wires are noted separately. The consistency of the results is shown in Fig. 4 where all the results on partly sheltered bronze screen at Jersey City have been assembled.

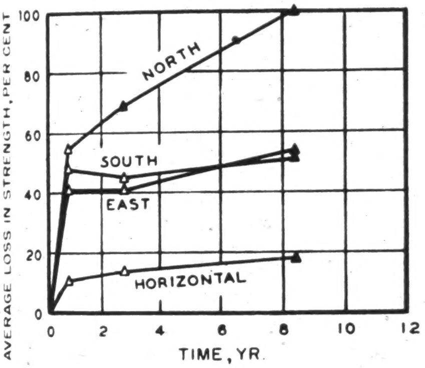

FIG. 5.—Box Exposures at Pittsburgh on 18-8 Steel.
Blacked-in points indicate some wire failures.

RESULTS OF TESTS

Box Exposures:

At Pittsburgh the 18-8 screens rapidly became covered with a thick, loose, sooty deposit. General corrosion was slight, but pitting was severe, particularly at intersections between the wires. As shown by Fig. 5 the pitting rate dropped off rapidly with time, and except at the north exposure, most pits were little deeper at 8 yr. than at 1 yr. However, more pits formed and some continued to grow until the wires were perforated.

The marked variation between the different directions of exposure is noteworthy, and indicates the importance of considering this factor. The screen exposed facing north corroded faster than the others, and the one exposed horizontally much slower. It is believed that this variation is accounted for by the wash of rain, the drying action of the sun, and the percentage of the time wet. Other results have shown that the deleterious effect of shelter is generally more important than exposure direction.

At North Brigantine the box exposures rested on sand, and the burnishing action of wind-blown sand was definitely beneficial. Nevertheless the screens did develop rust stains and shallow pits. The first wire failures were observed on screens facing south at 7.2 yr., at which time the screens were discarded. Results are in Table III.

TABLE III.—BOX EXPOSURES (BOLDLY EXPOSED).

18-8 steel screen, exposed in April, 1932. Shoot wires tested, vertical except on horizontal screen.

Location	Time, yr.	Average Loss in Strength, per cent				
		North	South	East	West	Horizontal
Pittsburgh	0.8	55	48	41		11
	2.8	69[a]	45	41		14
	8.4	100	51[a]	54[a]		18[a]
North Brigantine[b]	0.8	9	19		11	9
	2.8	1	6		16	10
	7.2		[a]			

[a] Some wire failures.
[b] At North Brigantine east was seaward.

1932 Tests:

At Bayonne the bronze screens became thickly encrusted with a dark green corrosion product, which decreased the visibility through the screen. Removing part of the corrosion product showed that it was the color of cuprous oxide next to the metal. The bronze itself corroded fairly uniformly. At 13 yr. there were no visible breaks in undisturbed screen, although wire failures were detected at 8 yr. by plucking out individual wires. Apparently the corrosion product held the screen together. Wires from the partly sheltered upper part of the screen corroded somewhat faster than wires from the lower unsheltered part, and the screen exposed facing north corroded faster than the one exposed facing south.

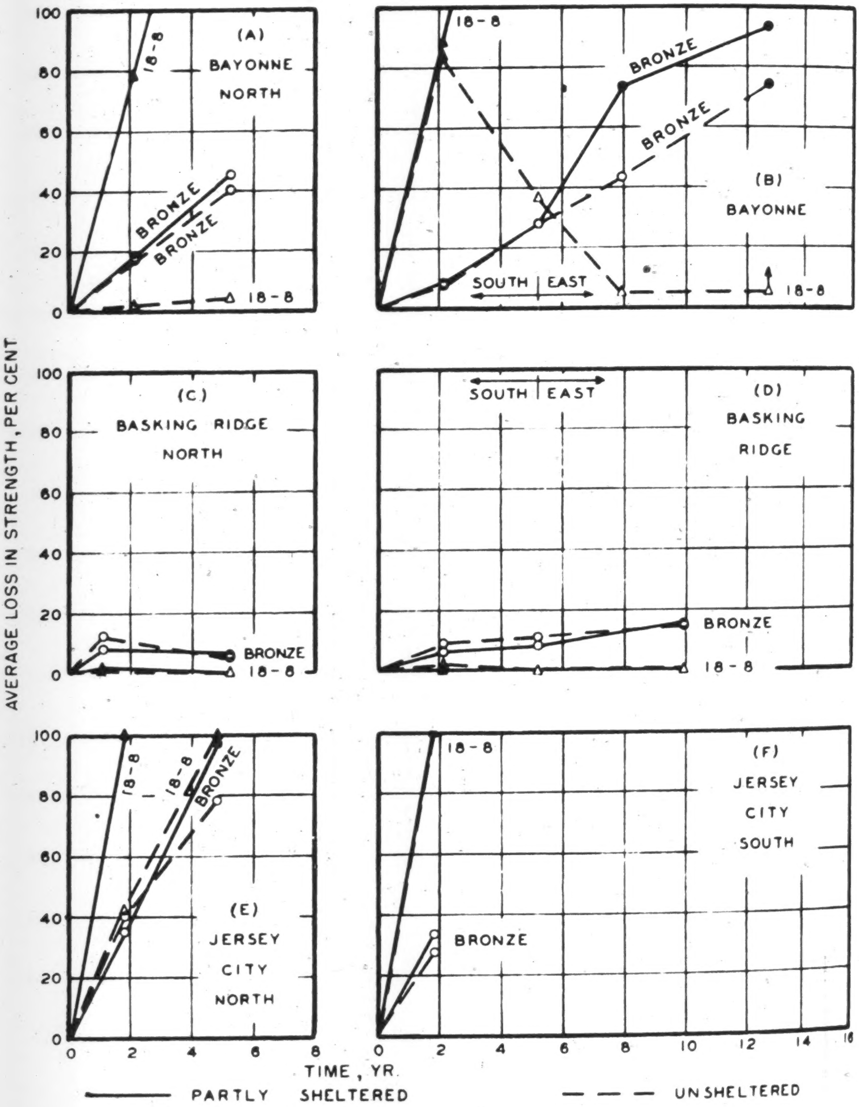

FIG. 6.—1932 Tests.

Blacked-in points indicate some wire failures.

In the case of 18-8 steel the difference in shelter had a big effect. The upper part was in poor condition after 2 yr.; whereas the lower part was in fairly good condition after 13 yr. with only a few sharp breaks. None of these broken wires happened to be included in the tension tests at 12.7 yr., although a disproportionate number were included at 2.1 yr. (see Fig. 6(*B*)). These screens had a low weight loss, the loss in strength being due to sharp pits. Much of the original surface was still present. On the racks the 18-8 steel screens were coated with a thin, loose, dark coating.

TABLE IV.—1932 TESTS.
Vertical shoot wires tested.

Location	Time, yr.	Average Loss in Strength, per cent							
		Bronze				18-8 Steel			
		North		South Then East[a]		North		South Then East[a]	
		Upper Partly Sheltered	Lower Un-sheltered	Upper Partly Sheltered	Lower Un-sheltered	Upper Partly Sheltered	Lower Un-sheltered	Upper Partly Sheltered	Lower Un-sheltered
Bayonne	2.1	18	17	8	7	78[b]	2	88[b]	83[b]
	5.2	45	40	28	28	100	4	100	37
	7.9			73[b]	43			100	5
	12.7			93[b]	74[b]			100	5[b]
Basking Ridge	2.1	8	12	6	9	2	1	0	2
	5.2	6	5	8	11	0	0	0	0
	9.9			15	14			0	0
Jersey City	1.8	35	40	33	27	100	42	100	100
	4.8	97[b]	78			100	100	100	100
Fire Island[c]	2.0	12	14			16	19	[b]	
	5.1	17	18			47	28		

[a] Changed to east at 5.2 yr.
[b] Some wire failures.
[c] Rack repeatedly knocked down and finally destroyed by storms; south was seaward.

TABLE V.—1933 TESTS (BAYONNE).

Screen Material	Time, yr.	Average Loss in Strength, per cent							
		Boldly Exposed Facing North				Completely Sheltered Facing South			
		Warp		Shoot		Warp		Shoot	
		Horizontal	Vertical	Horizontal	Vertical	Horizontal	Vertical	Horizontal	Vertical
Bronze	0.5	1	0	5	6	8	6	15	10
	1.0	4	4	5	5	3	3	8	5
	2.0	16	15	15	16	15	19	16	16
	6.4	45	46	65[a]	55	75[a]	56	95[a]	81[a]
	8.3					100	100	100	100
	11.1	56	60	65	76				
18-8 steel	0.5	7	4	5	4	27	11	30	14
	1.0	0	0	3	0	6	65[a]	52[a]	64[a]
	2.0	5	2	56[a]	2	100	100	100	100
	6.4	0	0	22	0				
	11.1	9	12	36[a]	11				

[a] Some wire failures.

At Basking Ridge, the 18-8 screens were substantially uncorroded through 10 yr. The screens were generally bright but had some faint rusty spots near the top. Bronze became a uniform dark green but corrosion was slow (Fig. 6 (*C*) and (*D*)).

At Jersey City, corrosion of both bronze and 18-8 was very rapid. Partly sheltered bronze lasted 4.8 and unsheltered bronze 6.5 yr. (Compare Figs. 4 and 8(*C*)). Partly sheltered 18-8 steel lasted less than 2 yr., and unsheltered not much longer. All the screens were very dirty at this location. Bronze corroded uniformly and 18-8 pitted.

At Fire Island the screens were so repeatedly damaged by storms that even the early results are not considered too significant. It seemed clear that in this marine atmosphere 18-8 was subject to severe pitting. For a summary of the 1932 tests, see Table IV.

1933 Tests:

The 1933 tests (see Table V) were conducted only at Bayonne and were aimed at clarifying further the effect of shelter and exposure conditions. A comparison of Figs. 6 and 7 shows that the tests confirmed that sheltered 18-8 at Bayonne could be expected to have perforations in every wire by about 2 yr., and that exposed screen could be expected to last a long time with only a few sharp pits and an occasional wire perforation. The tests also confirmed the deleterious effect of shelter on bronze screen. Completely sheltered bronze lasted 8.3 yr., and boldly exposed screen was doing better than partly sheltered screen.

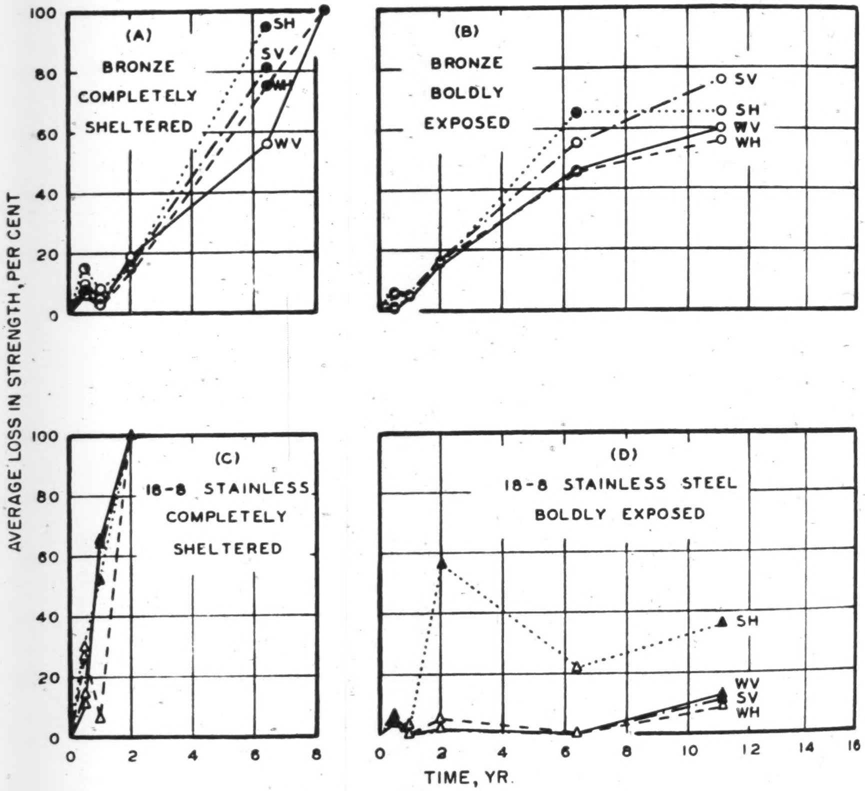

Fig. 7.—1933 Tests at Bayonne.

Blacked-in points indicate some wire failures.

W = Warp wires *V* = Vertical wires
S = Shoot wires *H* = Horizontal wires

TABLE VI.—1935 TESTS.

North Brigantine tests. Vertical wires tested. Screens faced east or seaward.

Material	Time, yr.	Average Loss in Strength of Partly Sheltered Specimens, per cent
Type 316 molybdenum steel	4.5 6.4	13 24[a]
Type 302 corrosion-resistant steel	3.5	[a]

[a] Some wire failures.

As compared with shelter, other conditions of exposure seemed of minor importance. In the case of bronze, hard-drawn shoot wires corroded somewhat faster than the slightly softer warp wires. There was no clear-cut difference between the behavior of the horizontal and vertical wires of the screens.

1935 Tests:

In 1935 a direct comparison was started between type 302 and type 316 steel screen in the marine atmosphere at North Brigantine (see Table VI). The screens were exposed partly sheltered. The type 302 screen quickly developed a nonuniform rusty appearance. Examination with a magnifying glass showed deep undercutting pits. Wire failures were evident by $3\frac{1}{2}$ yr.

The type 316 molybdenum corrosion-resistant steel screen performed much better. It did develop faint scattered rust spots which were more evident from the sheltered side of the screen. These were caused by scattered pits, most of which were shallow, but a few of which were deep. By 6.4 yr. five barely noticeable wire failures were detected. At this time the average loss in strength was 24 per cent. Subsequently this exposure test was destroyed by coastal storms.

1937 Tests:

In these tests (see Table VII and Fig. 8) a second lot of type 316 molybdenum corrosion-resistant steel screen was exposed at four locations in direct comparison with bronze. The type 316 screen was exposed as-received and as-passivated by a 20-min. immersion in 25 per cent nitric acid at 145 F. Judging from the test results the passivating treatment was not needed.

The marine Fire Island test was lost in the hurricane of 1938. Inspections at 3 and 12 months showed faint rust spots and streaks on the type 316 screens, which were more noticeable on the upper partly sheltered portions.

At the other locations there was no real corrosion of the type 316 screens through 8 yr. Such performance in industrial and sulfurous atmospheres is phenomenal. The screens collected loose

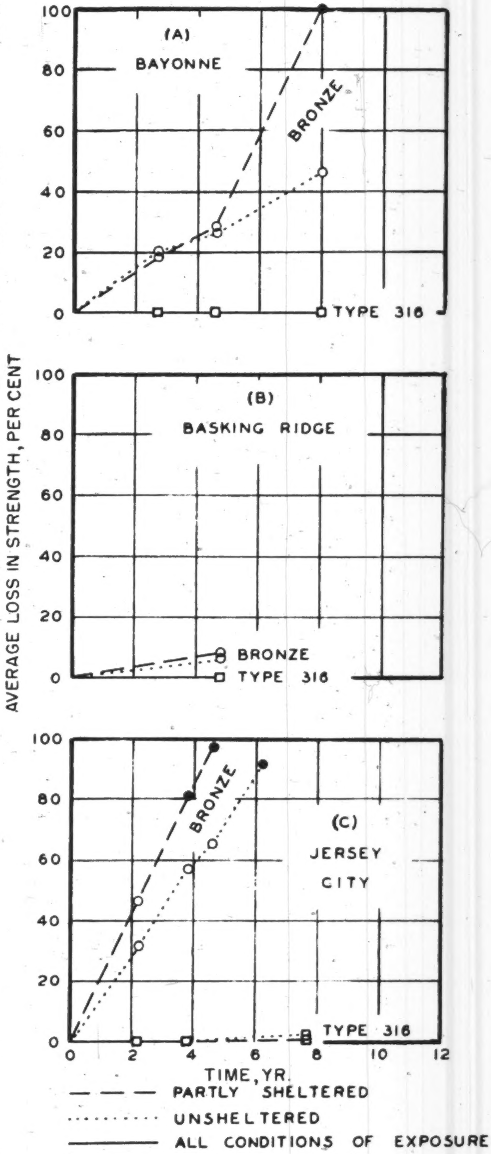

FIG. 8.—1937 Tests.
Blacked-in points indicate wire failures.

TABLE VII.—1937 TESTS.

Vertical warp wires tested. Screens faced north at Jersey City, west at other locations, except that at Bayonne boldly exposed screen faced south and completely sheltered north.

Location	Time, yr.	Average Loss in Strength, per cent									
		Bronze		Type 316 Molybdenum Steel							
				As-Received				Passivated in HNO_3			
		Partly Sheltered Rack		Partly Sheltered Rack		Boldly Exposed	Completely Sheltered	Partly Sheltered Rack		Boldly Exposed	Completely Sheltered
		Upper Partly Sheltered	Lower Unsheltered	Upper Partly Sheltered	Lower Unsheltered			Upper Partly Sheltered	Lower Unsheltered		
Bayonne	2.7	18	20	0	0	0	0	0	0	0	0
	4.6	28	26	0	0	0	0	0	0	0	0
	8.0	100	46	0	0	0	0	0	0	0	0
Basking Ridge	4.8	8	6	0	0			0	0		
Jersey City	2.2	46	31	0	0			0	0		
	3.8	81[a]	57	0	0			0	0		
	4.6	97[a]	65								
	6.2		91[a]								
	7.7			0	1			1	4		

[a] Some wire failures.

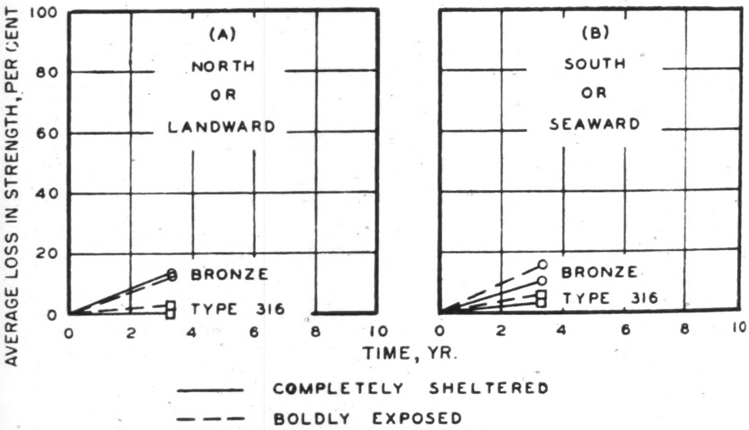

FIG. 9.—1941 Tests at Block Island.

TABLE VIII.—1941 TESTS.

Block Island tests. Vertical warp wires tested.

Material	Time, yr.	Average Loss in Strength, per cent			
		North or Landward		South or Seaward	
		Boldly Exposed	Completely Sheltered	Boldly Exposed	Completely Sheltered
Bronze	3.3	12	13	15	10
Type 316 molybdenum steel	3.3	3	0	6	3

dirt, but this could easily be rubbed off leaving the screens bright.

The behavior of bronze screen confirmed earlier results. The wire failures at 8 yr. were confined to the upper 2 or 3 in. of the screen which was practically completely sheltered.

1941 Tests:

These tests at Block Island (see Table VIII and Fig. 9) were intended to supplement earlier marine atmospheric tests which had been lost in storms. As at the other marine sites sheltered molybdenum stainless steel insect screen developed faint scattered rust spots in 1 yr., and by 2 yr. unsheltered screen also had faint rust spots. Nevertheless these screens remained predominantly bright. Tension tests at 3.3 yr. showed only slight loss in strength. Examination at low magnification showed an occasional shallow pit.

As expected the bronze screen became dark green and was corroding uniformly. The tests had not continued long enough to tell much about the effect of shelter or exposure direction. It is quite possible that this marine site was not quite as corrosive as the earlier ones which were located closer to the surf.

TABLE IX.—1943 TESTS.

Vertical warp wires tested. Screens faced south.

Location	Time, yr.	Average Loss in Strength, per cent			
		Bronze		Type 316 Molybdenum Steel[b]	
		Completely Sheltered	Partly Sheltered	Completely Sheltered	Partly Sheltered
Bayonne	2.0	22		9	
Jersey City	1.5		28		26[a]
Block Island	1.8	10		0	

[a] Some wire failures.

[b] Screen contains some 18-8 steel wires.

1943 Tests:

In 1943 another series of tests (see Table IX) was started comparing a third lot of molybdenum steel screen with bronze. Unfortunately it soon developed that through error a few regular 18-8 warp wires had been woven into the corrosion-resistant steel screen. This was established by qualitative analyses. The 18-8 wires corroded rapidly and were completely gone at Bayonne in 1 yr. and at Jersey City at the first inspection at 1.5 yr. They were corroding rapidly at Block Island also, but showed no perforations through 1.8 yr.

At Block Island the presence of the 18-8 wires did not harm the molybdenum steel screen, there being no loss in strength of the type 316 wires through 1.8 yr. However, at Jersey City and Bayonne it is feared that iron corrosion products from the 18-8 wires destroyed the passivity of the wires containing molybdenum. It is difficult otherwise to account for the absence of corrosion in the 1937 tests and the deep pitting in the 1943 tests.

EVALUATION OF TEST RESULTS

The tests established that bronze screen corrodes uniformly, while if corrosion occurs on corrosion-resistant steel screen, it is in the form of pits. Regular 18-8 does not seem suitable as an all-purpose screen because it is susceptible to rapid pitting in corrosive atmospheres particularly where sheltered. In the 1937 tests corrosion-resistant steel containing 3 per cent molybdenum showed phenomenal resistance to corrosion in sulfurous, industrial, and rural atmospheres. The material used for the 1943 tests contained only 2.1 per cent molybdenum. Its behavior casts some doubt on the resistance of type 316 steel screens in sulfurous atmospheres, but it seems likely that the corrosion in these tests can be accounted for.

There is also some doubt about the behavior of molybdenum steel screen in marine atmospheres. As yet there is no good comparison of the relative durability of bronze and type 316 screen in marine atmospheres, although in due time the 1941 and 1943 tests should fill this gap. It seems certain that molybdenum steel screen will develop faint rust spots and shallow scattered pits in marine exposures.

It is pertinent that recently the laboratory had the occasion to examine a type 316 screen that had been in use at Miami Beach, Fla., for an unknown length of time. There was severe local pitting with some wire failures, although the weight loss was low. The pitting led to the formation of rust streaks. The rust in turn spread the pitting with the result that pits usually occurred in clusters and that wires were often pitted near the intersections with a rusty wire. This screen analyzed 0.10 per cent carbon, 10.69 per cent nickel, 17.50 per cent chromium, and 2.66 per cent molybdenum.

All this makes it seem probable that the useful life of type 316 screens in marine atmospheres will not be significantly greater than that of bronze. The development of light rust spotting in sheltered areas would be expected to lead to customer complaints.

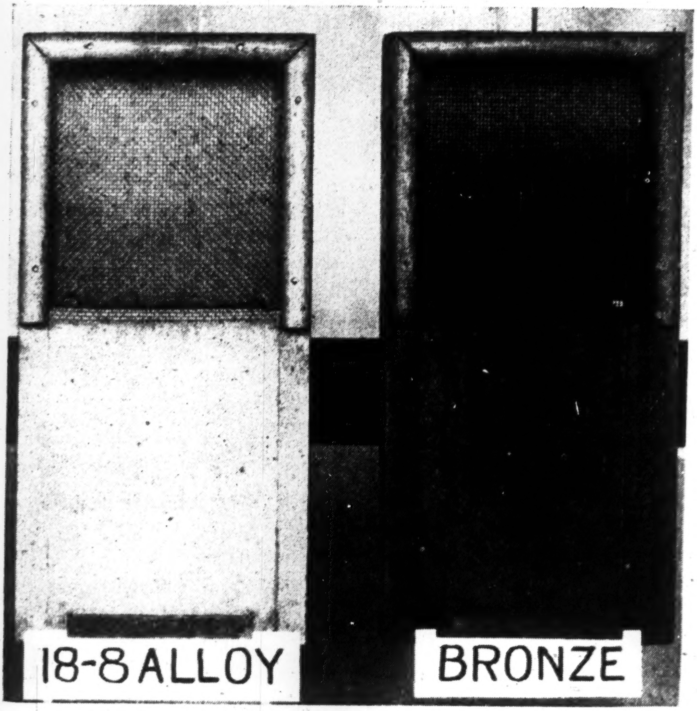

Fig. 10.—Staining of Paint by Corrosion Products Draining from Screens.

ADVANTAGES OF CORROSION-RESISTANT STEEL SCREEN

Aside from the question of durability, corrosion-resistant steel has some important advantages over bronze screen. It is well known that the corrosion product draining down from bronze screen will stain white paint, particularly paints high in sulfides. Corrosion-resistant steel screen would be expected to be well nigh nonstaining, especially since its weight loss is low. This was shown by some tests in which small pieces of screen were tacked above wood freshly painted with white lithopone paint, and exposed unsheltered at Bayonne. Within a short time the paint underneath the bronze screen was badly stained, whereas that under the corrosion-resistant steel screen was still white. The condition of the panels after 4 months is shown in Fig. 10.

Another advantage is that corrosion-resistant steel screen is strong. It can be made with much greater strength than any other screen on the market.

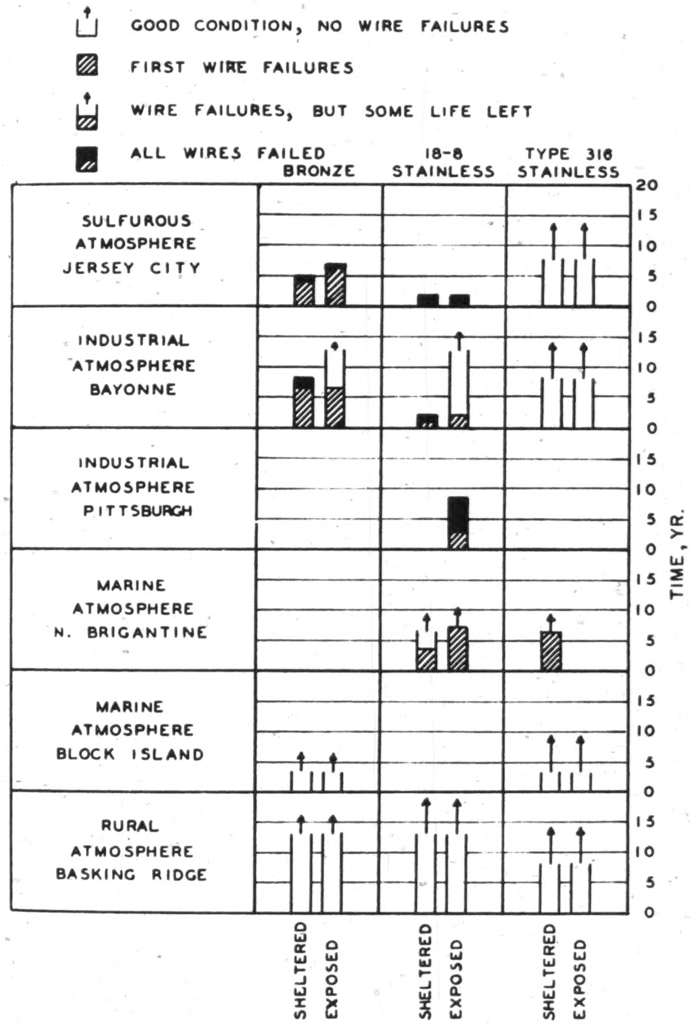

Fig. 11.—Summary Showing Minimum Lives of Screens.

A third advantage is clearer vision. The wires are of smaller diameter than on the bronze screen, which lets in more air. Also the corrosion products on bronze screen form a thick encrustation which cuts down visibility. Many bronze screens were observed with pores completely plugged with the accumulation of dirt and corrosion product. Bronze screens are difficult to clean because of the adherent corrosion products whereas type 316 stainless steel screens are readily washed clean.

The corrosion results to date can be summarized by a diagram showing the time in years to the first occurrence of wire failures and to complete failure as is done in Fig. 11.

DISCUSSION

MESSRS. J. R. FREEMAN, JR.[1] AND A. W. TRACY[1] (*presented in written form*).—We have made exposure tests on a number of screen materials and have found none significantly superior to commercial bronze in resistance to atmospheric corrosion. Contrary to the results obtained by Messrs. Wesley and Copson, we have found the corrosion resistance of commercial bronze to be somewhat better in industrial atmosphere than in marine atmosphere. This discrepancy in test results probably only emphasizes the fact that there is a wide variation in corrosiveness among both industrial and marine exposures.

TABLE X.—COMPOSITIONS OF SCREENS

Material		Composition, per cent						
		Copper	Zinc	Silicon	Manganese	Nickel	Iron	Other Elements
Copper	Warp Shoot	99.961	....	....	....	....	....	....
Bright Commercial Bronze	Warp Shoot	90.55	9.43	....	....	....	0.02	....
Antique Bronze	Warp	90.45	9.52	....	....	....	0.02	0.01 Pb
	Shoot	89.77	10.21	....	....	....	0.01	0.01 Pb
Everdur	Warp	95.83	0.00	3.02	0.96	....	0.09	....
	Shoot	95.71	0.00	2.87	1.12	....	0.09	....
Ambrac	Warp Shoot	73.66	5.78	....	0.48	20.00	0.08	....
Monel	Warp	26.86	....	....	2.14	68.37	2.56	....
	Shoot	26.75	....	....	2.15	69.18	1.88	....
Inconel	Warp	....	....	....	....	80.36	6.40	13.24 Cr
	Shoot	....	....	....	....	79.68	6.68	13.64 Cr
Phosphor Bronze	Warp	98.18	....	....	....	....	....	1.83 Sn
	Shoot	98.07	....	....	....	....	....	1.70 Sn

In our tests, screens 2 ft. square were exposed on unsheltered wooden frames in a vertical position. The accompanying Fig. 12 shows the industrial location on the roof of a mill building in Waterbury, Conn. The 2-ft. square screens are on the racks in the foreground and screen samples fastened over painted wooden panels for paint-staining tests are in the background. Figure 13 is a view of the marine location situated on a small island in Long Island Sound about four miles off shore. A rural test site is shown in Fig. 14. Only screens over painted wooden panels were exposed at this place which is about 30 miles northwest of Waterbury, Conn.

The accompanying Table X gives the compositions of the 2-ft. square screens.

Table XI summarizes the results of tests on the 2-ft. square screens in industrial and marine atmospheres. A screen was judged to have failed when

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[1] Technical Manager and Assistant Metallurgist, respectively, The American Brass Co., Waterbury, Conn.

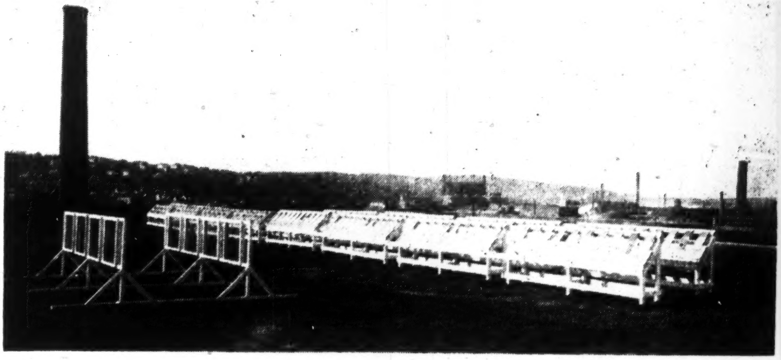


FIG. 12.—Screens and Painted Panels Exposed to Industrial Atmosphere at Waterbury, Conn.

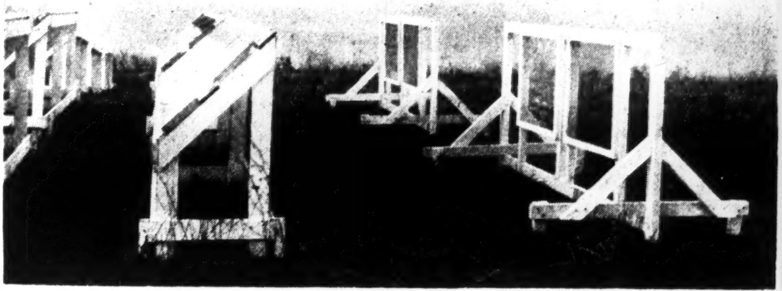


FIG. 13.—Screens and Painted Panels Exposed to Marine Atmosphere on Island in Long Island Sound.

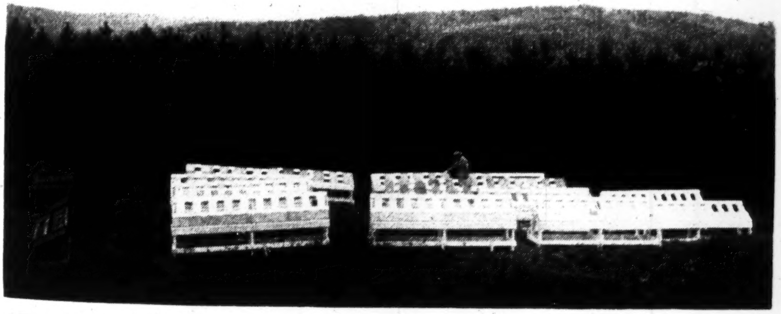
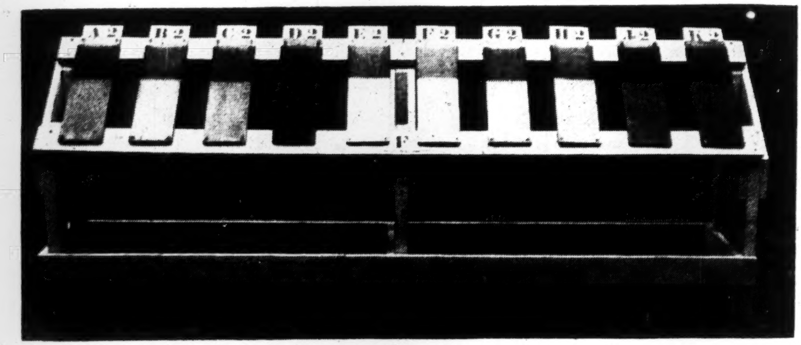
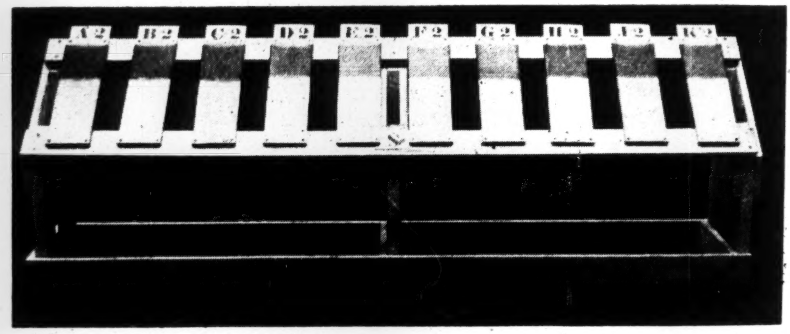


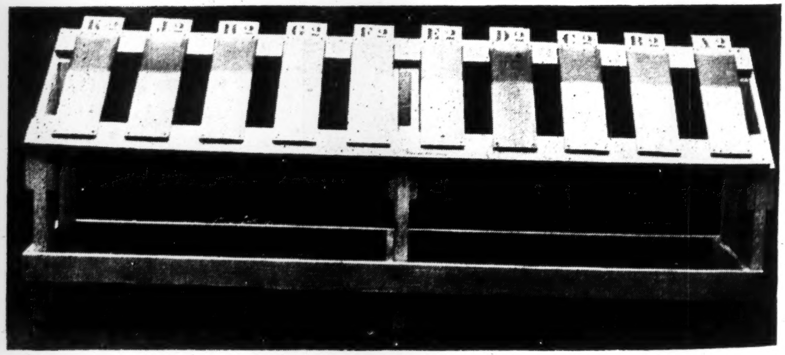
FIG. 14.—Painted Panels Exposed to Rural Atmosphere at Winchester, Conn.



(*a*) **Exposed to industrial atmosphere.**



(*b*) **Exposed to marine atmosphere.**



(*c*) **Exposed to rural atmosphere.**

FIG. 15.—Painted Panels Under Commercial Bronze Screen Exposed 2 yr.

A and B	**100 per cent white lead**
C	**50 per cent white lead** **50 per cent zinc oxide**
	100 per cent zinc oxide **40 per cent white lead**
	30 per cent barium sulfate **20 per cent zinc oxide** **10 per cent titanium oxide**
F	**45 per cent barium sulfate** **35 per cent zinc oxide** **20 per cent titanium oxide**
G	**90 per cent basic lead sulfate** **10 per cent zinc oxide**
H	**50 per cent basic lead sulfate** **50 per cent zinc oxide**
J	**Lithopone**
K	**100 per cent zinc oxide enamel**

holes or tears occurred that were considered large enough to permit passage of house flies.

Failure of the Ambrac and monel screens was due to general thinning of the wires. Failure of the other screens was usually due to pitting. It will be noted from a consideration of Table XI that none of the other screens was significantly superior to commerical bronze at either the industrial or marine location. None of the meshes of the screens became plugged with corrosion products at either of the test sites.

Figure 10 in the Wesley and Copson paper shows a bronze screen over a wooden panel, painted with lithopone paint, and the paint shows severe staining. This type of paint is quite readily stained by iron rust or copper salts. We have made a large number of paint-staining tests. Panels painted with various types of paint were exposed to industrial weathering for 2 yr. at the site in Fig. 12. The panels were exposed at an angle of 45 deg. from the horizontal and facing south. The panels are 18 by 6 by ½ in. A piece of 16-mesh commercial bronze screen is wrapped around the panel near the upper end so as to cover an area of about 6 by 6 in. Figure 15(*a*) is a photograph taken outside the laboratory building after the panels were removed from the test site. The letters at the top of the panels identify the type of paint used (see figure caption).

It will be noted that severe staining has taken place on the paints high in zinc pigments, very little staining has occurred on the white lead or basic lead sulfate paints, and no staining has occurred on the paints containing titanium oxide.

Figure 15(*b*) represents another group of the same type of specimens exposed 2 yr. to marine atmosphere. Only the lithopone-painted panel is badly stained.

Figure 15(*c*) represents similar specimens exposed to rural atmosphere for 2 yr. Here again only the high-zinc pigmented paints are stained.

TABLE XI.—LIFE OF 16-MESH SCREENS EXPOSED TO INDUSTRIAL AND MARINE ATMOSPHERES.

Material	Industrial Atmosphere			Marine Atmosphere		
	Date of Exposure	Time to First Wire Break, yr.	Failed, yr.	Date of Exposure	Time to First Wire Break, yr.	Failed, yr.
Copper	Dec. 1928	9½	9¾	Nov. 1928	6	7⅓
Bright Commercial Bronze	Dec. 1928	No breaks in 17 yr.		Nov. 1928	8½	9½
Antique Bronze	Dec. 1928	15½	16½	Nov. 1928	....	7⅛
Everdur	Dec. 1928	14½	15	Nov. 1928	6	9
Ambrac	Dec 1928	9½	15	Nov. 1928	6½	8½
Monel	Dec. 1928	....	6	Nov. 1928	....	5⅛
Phosphor Bronze	Jan. 1929	No breaks in 17 yr.		Jan. 1929	11½	13½
Inconel	June 1935	4	7⅛	June 1935	5⅛	9

MR. V. V. KENDALL.[2]—Did you have any 18 per cent chromium, 8 per cent nickel alloy screens to compare with the other metallic materials?

MR. TRACY.—No. Our tests were started around 1930 and no 18 per cent chromium, 8 per cent nickel alloy screening was available at that time.

MR. KENDALL.—Is the commercial bronze you tested of the approximate composition of the bronze the authors tested?

MR. TRACY.—It is the same approximate composition.

MR. KENDALL.—Why the difference in the stain? The authors show stains by the commercial bronze and not by the 18-8 alloy (Fig. 10).

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[2] National Tube Co., Pittsburgh, Pa.

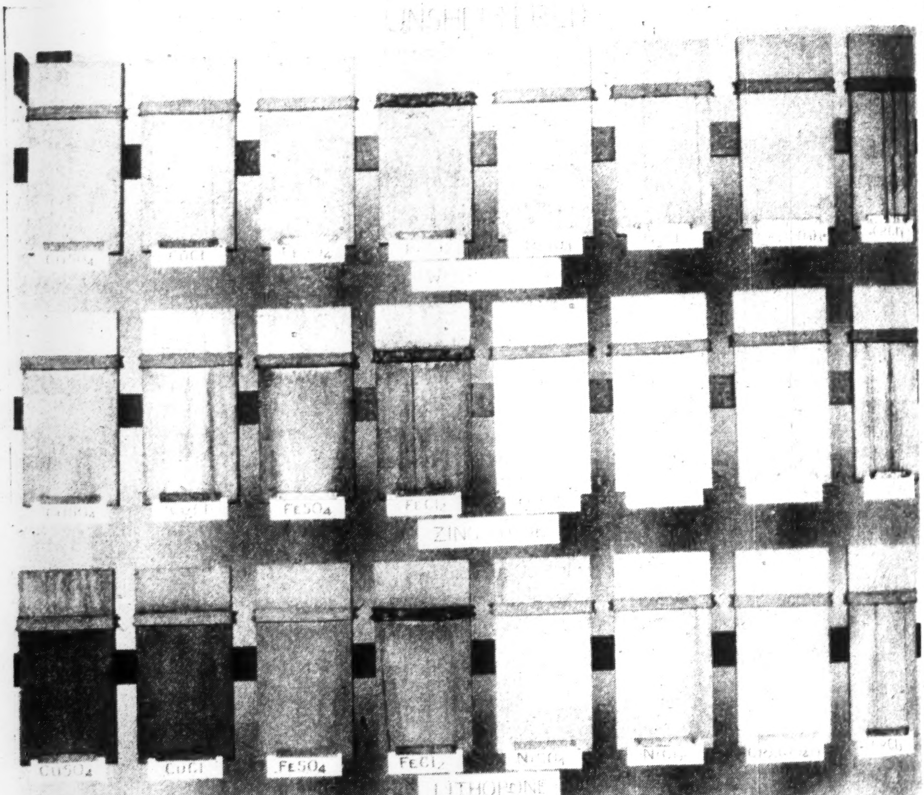
CuSO4
FeSO4
FeCl2
ZINC
CuSO4
CuCl
FeSO4
FeCl2
NiSO4
NiCl2
LITHOPONE
CuSO4
CuCl
FeSO4
FeCl2
NiSO4
TITANIUM OXIDE
CuSO4
CuCl
FeSO4
FeCl2
NiSO4
NiCl2
Cr2(SO4)3
CrCl3
READY MIXED

FIG. 16.—Staining of White Paints by the Wash from Metallic Salts.

MR. H. R. COPSON (*author*).—The discussion presented by Messrs. Freeman and Tracy comprises a valuable addition to our knowledge of the behavior of insect screen materials. As brought out by Mr. Kendall, Freeman and Tracy did not test "stainless" steel insect screens. It was the purpose of the paper to call attention to this screen material and to show that it did have some distinct advantages over commercial bronze.

The screens exposed by Freeman and Tracy appear to have been tested only in a boldly exposed manner. As pointed out in the paper, shelter greatly accelerates wire failures. This difference in testing procedure may account in part for their reverse rating of the relative corrosivity of marine and industrial atmospheres with respect to bronze screen. We believe that testing only boldly exposed screen is apt to be misleading if the results are used to predict screen life in general household service.

Freeman and Tracy appear to have relied on visual examination to detect failures, whereas in the paper great reliance was placed on tension tests. The two methods can give greatly different results. In the 1932 tests at Bayonne, there were no visible breaks in undisturbed bronze screen through 13 years, although individual wire failures were detected at 8 years by plucking out individual wires. Apparently in the case of bronze screen, thick layers of corrosion product obscure breaks and hold the screen together. The importance of this is difficult to evaluate. The undisturbed screen would certainly keep out house flies, but it was weak and would not stand any mechanical abuse such as might be inflicted by children, dogs, or the customary handling in spring and fall.

In both sets of tests the screens were exposed continuously. The usual practice of storing insect screens in a dry place during winter months would be expected to greatly increase service life.

The remarks made by Freeman and Tracy with regard to the effect of paint formulation on staining are well taken, and confirm conclusions drawn at this laboratory on the basis of extensive tests by G. L. Cox of our company under the direction of W. A. Wesley. The significant point is that a lithopone paint is about the most easily stained, and as shown by Fig. 10 the wash from stainless steel screen produced no staining of this paint. House paints are not usually selected with staining in mind, and unsightly stains beneath bronze screen are a common occurrence on newly painted houses. "Stainless" steel screens should eliminate any chance of this disfigurement.

With regard to the tests made by G. L. Cox, not only were screen samples mounted above painted panels, much in the manner of Freeman and Tracy, but the effect of individual corrosion products was studied. In this connection, Fig. 16 may be of interest. Painted wooden panels, 5 by 10 by $\frac{3}{4}$ in., were wrapped with narrow canvas bags containing metallic salt crystals, and exposed vertically at Bayonne. Copper and iron salts were particularly active in producing staining.

In answer to the question by Mr. Kendall, the reason there was no staining under the "stainless" steel screen in Fig. 10 is simply that there was practically no weight loss and hence no corrosion product.

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[9] Reading Steel Casting Div., American Chain and Cable Co., Inc., Reading, Pa.

MR. L. W. HOPKINS.[3]—I should like to ask Mr. Copson whether his 18 per cent chromium, 8 per cent nickel alloy screens were passivated.

MR. COPSON.—On the type 316 screens we ran a comparison between screens which were passivated in nitric acid and some which were as-received and it did not make any difference that we could see. Of course the type 316 screens did not corrode much anyhow. I am not sure about the 18-8 screens, whether they were passivated or not. I do not think it would make any difference. These were long-time exposures and the effect of any initial passivation would wear out.

RESULTS OF 15 YEARS' EXPOSURE TESTS ON CORROSION-RESISTANT STEELS*

By I. V. Williams[1] and K. G. Compton[1]

Synopsis

This paper discusses the results of the exposure of ten corrosion-resistant steels to New York City atmosphere for 15 yr. These steels represented five different alloys and were manufactured by four different companies. The loss in tensile strength of all alloys was small. The 13 per cent chromium steels showed considerable surface rust. The 18 per cent chromium, 8 per cent nickel and the 17 per cent chromium steels rusted slightly.

In 1928 it was decided to conduct an investigation of the resistance to atmospheric corrosion of several corrosion-resistant irons and steels which were commercially available. There was no information available at that time as to the actual corrosion resistance of these alloys under normal long-time exposures. Since then the use of corrosion-resistant steel has become very widespread and considerable experience in their use has been obtained. The purpose of this paper is to describe the tests that were conducted on the corrosion-resistant steels and to evaluate their corrosion resistance.

Test Program

As a preliminary step to the major exposure program, a group of sheet corrosion-resistant steels was selected to determine the proper surface preparation for the samples so that a uniform result could be obtained and so that the most favorable surface conditions could be selected. Samples of these materials were treated by the following surface preparation methods: sandblasting, grinding, pickling, pickling followed by light sandblasting, pickling followed by polishing, and scratch brushing.

The pickling was done in a 3 to 5 per cent nitric acid solution to which 1 per cent of hydrochloric acid had been added. These preliminary samples were exposed to a modified outdoor exposure test for several months. At the end of that time it was found that those samples which had been pickled and then given a light sandblast showed the best condition, and this surface preparation method was chosen for all of the samples covered by this paper.

These tests were started in 1930. Five different types of corrosion-resistant steels were tested, representing four different manufacturers and totaling ten steels in all. The chemical analysis and identification of these alloys are shown in Table I.

The steels were evaluated by examination of the condition of the surface, by weight-loss measurements, and by determination of the loss of tensile strength and elongation. For the surface examination and weight-loss determination, sample sheets 9 by 12 in. were provided while standard A.S.T.M. sheet tension specimens (Fig. 3, Standard Methods of Tension Testing of Metallic Materials (E 8 - 42)[2]) were used for the determination of the loss in physical properties. These samples were divided into three groups; one group referred to as the shelf-life specimens was kept indoors in a storeroom to be used as check specimens so that any changes in physical properties due to aging could be segregated from results of the corrosion tests. The other two groups were exposed to the atmospheric corrosion on roofs in New York City.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

[1] Members of Technical Staff, Bell Telephone Laboratories, Inc., New York, N. Y.

TABLE I.—ANALYSIS AND IDENTIFICATION OF CORROSION-RESISTANT STEELS.

Alloy	Composition, per cent		
	Nickel	Chromium	Carbon
1A	7.51	18.66	0.17
1B	9.49	17.55	0.27
1D	9.04	16.40	0.15
2A	0.16	16.66	0.15
3A	0.22	12.30	0.16
3B	0.12	12.49	0.062
3C	0.04	12.37	0.042
4B	0.12	13.31	0.46
4C	0.12	12.25	0.41
5B	23.83	8.60	0.12

The atmosphere in this location was found to be highly industrial in character and severely corrosive; the sulfur dioxide content is considerably above that normally found in city atmospheres and a relatively high percentage of dust is present. One group of specimens which was exposed on a roof 50 ft. above street level was also subjected for part of the exposure period to an additional simulated rainfall. This spray was supplied three times daily for 15-min. periods during 8 months, from April to December. After 8 yr. of the 15-yr. test, this spray was discontinued, since results on tests up to that time had shown no difference in the corrosion rate of these steels. The remaining specimens were exposed on a roof 150 ft. above the street level and were not subjected to the simulated rainfall. After 10 yr. of the test, all of the specimens were consolidated on this higher location.

The 13 per cent chromium type corrosion resistant steels were all tested in the heat-treated condition. These alloys were heat-treated at 1800 F., quenched in oil, and drawn at 900 F. for $\frac{1}{2}$ hr. All of the other alloys were tested in the as-received condition.

The tension values were determined on an Amsler 20,000-lb. testing machine using a maximum free head speed of 0.25 in. per inch of gage length per minute. Rockwell hardness tests were made on the hardened materials using the C scale. The B scale was used for the other alloys. Ten specimens from each alloy were used for the initial tests. Five specimens of each material were used for the 1, 10, and 15-yr. tests. Only one or two specimens were tested at the 2- and 3-yr. intervals to determine whether any appreciable change had occurred. Six of the 9 by 12-in. panels were exposed at the same time as the tension specimens. The panels were weighed before being exposed. These panels were weighed every six months during the first three years. The weighings were then discontinued since no appreciable weight loss was recorded. At the end of the 15-yr. period, three of the panels were weighed as removed from the roof and were then stripped of corrosion products in an inhibited hydrochloric acid solution and reweighed. The stripping process continued until a constant weight was obtained. One alloy, No. 5B, did not lend itself well to this method in that the base metal seemed to flake off under the influence of the acid. Sandblasting was resorted to, although this method did not yield entirely satisfactory results. As a result, the corrosion values given for this alloy are problematical, particularly in view of the slight loss in physical properties.

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[2] 1944 Book of A.S.T.M. Standards, Part I, p. 962.

PHYSICAL TESTS

Physical tests were made on the tension specimens at the end of 1, 2, 3, 10, and 15 yr. When this test program was originally planned, tests were to have been made each year over a 5-yr. period. However, so little changes occurred at the end of 3 yr. that the program was continued for 15 yr.

TABLE II.—MECHANICAL PROPERTIES OF CORROSION-RESISTANT STEELS.

		Initial			1 yr.		2 yr.		3 yr.		10 yr.		15 yr.	
Alloy	Exposure	Ultimate Tensile Strength, psi.	Elongation in 2 in., per cent	Rockwell Hardness	Ultimate Tensile Strength, psi.	Elongation in 2 in., per cent	Ultimate Tensile Strength, psi.	Elongation in 2 in., per cent	Ultimate Tensile Strength, psi.	Elongation in 2 in., per cent	Ultimate Tensile Strength, psi.	Elongation in 2 in., per cent	Ultimate Tensile Strength, psi.	Elongation in 2 in., per cent
1A	Shelf life	110 100	66	B98							100 700	60	101 300	58
	50-ft. roof				107 500	....	111 000	....	106 600	62	103 000	57		
	150-ft. roof				105 100	....	110 300	....	107 700	60	100 500	56	104 200	57
1B	Shelf life	108 100	64	B98	106 600	....			108 000	63	111 700	55	114 500	57
	50-ft. roof				105 600	56	108 900	....	105 700	62	102 900	59		
	150-ft. roof				106 400	59	108 200	....	105 300	60	103 000	58	103 800	60
1D	Shelf life	119 700	74	B99					120 500	65	101 000	65	102 300	60
	50-ft. roof				104 000	67.0	119 400	....	105 100	75	102 400	62		
	150-ft. roof				100 400	68.0	117 500	....	119 600	59	107 400	66	99 300	64
2A	Shelf life	75 200	28	B91	....	....	....	....	....	....	74 500	27	74 100	27
	50-ft. roof										74 200	26		
	150-ft. roof										74 900	27	73 900	27
3A	Shelf life	214 200	7.5	C48							206 900	6.7	201 100	8.0
	50-ft. roof				206 600	6.0	215 100	....	211 600	7.0	211 200	7.4		
	150-ft. roof				208 900	8.0	213 300	....	211 500	8.0	210 700	6.5	209 900	6.3
3B	Shelf life	171 800	7.0	C44	166 600	....			166 800	8.0	172 400	9.6	171 400	8.8
	50-ft. roof				170 000	9.0	166 000	....	170 200	8.0	170 000	8.0		
	150-ft. roof				177 900	9.0	186 600	....	176 100	8.0	174 400	9.4	163 800	8.0
3C	Shelf life	175 700	7.7	C39	179 300	9.5			177 000	7.5	173 500	8.0	177 000	8.3
	50-ft. roof				176 000	8.0	180 800	....	167 600	7.0	172 500	8.0		
	150-ft. roof				175 000	....	178 000	....	180 300	10.0	174 000	8.7	174 500	7.4
4B	Shelf life	216 900	3.0	C48							211 100	2.5	207 000	3.0
	50-ft. roof						207 700	....	214 400	3.0	207 000	2.1		
	150-ft. roof				215 000	3.0	202 500	....	217 100	....	205 400	1.5	207 000	3.0
4C	Shelf life	225 100	4.0	C49					218 500	2.0	226 300	5.5	222 400	3.6
	50-ft. roof				217 200	....	221 000	....	236 000	4.0	208 200	1.3		
	150-ft. roof				221 600	9.0	214 000	....	238 000	....	215 200	2.3	206 500	1.8
5B	Shelf life	102 900	33	B98	97 700	....			98 700	34	95 600	32	96 200	30
	50-ft. roof				95 300	....	97 500	....	98 700	33	94 600	32		
	150-ft. roof				100 300	33	99 300	....	100 700	34	96 400	33	90 600	32

TEST DATA

The composition and identification of the materials tested are shown in Table I. Table II shows the physical properties obtained on the materials as tested. The weight-loss measurements are shown in Table III and are expressed both in terms of milligrams loss per square inch per 15 yr. as well as thousandths of an inch penetration per 15 yr. No attempt has been made to express corrosion in terms of weight loss per unit area or penetration per year, since this would assume corrosion at a uniform rate, which in all likelihood would be an incorrect assumption. The appearance rating of the alloys after 15 yr. is indicated in Table IV.

From the visual examination of the materials, alloys 1A, 1D, and 2A were excellent from the standpoint of resistance to corrosion and are slightly superior to the other steels. Alloy 5B has the poorest corrosion resistance of any of the steels tested. The remaining steels are slightly less corrosion-resisting than alloy 1A and fall in approximately the same grouping with the exception of alloy 4B which is slightly inferior.

TABLE III.—WEIGHT-LOSS MEASUREMENTS

Alloy	Sample	Weight, g.					Loss per 15 yr., mg. per sq. in.	Penetration per 15 yr., in.
		Original	3 yr.	15 yr.	15 yr. Stripped	Loss		
1A............	No. 4	835.97	835.97	836.9	836.5	0.3	1.4	0.000011
	No. 11	816.78	816.77	817.6	817.2	0.4	1.9	0.000014
1B............	No. 1	888.36	888.40	888.9	888.1	0.3	1.4	0.000011
	No. 8	911.88	911.89	912.5	911.5	0.4	1.9	0.000014
	No. 19	850.55	850.45	850.0	850.0	0.6	2.8	0.000021
1D............	No. 1	841.58	841.54	842.0	840.7	0.9	4.2	0.000032
	No. 13	841.58	841.57	842.1	841.5	0.1	0.5	0.000004
	No. 17	840.48	840.44	841.0	840.0	0.5	2.3	0.000018
2A............	No. 1	888.79	888.80	889.5	889.8	0.0	0.0	0.000000
	No. 2	885.35	885.35	885.9	885.2	0.2	0.9	0.000007
	No. 3	861.64	861.65	862.5	861.4	0.2	0.9	0.000007
3A............	No. 2	729.33	729.29	729.5	727.7	1.6	7.4	0.000058
	No. 9	752.10	752.10	752.3	750.5	1.6	7.4	0.000058
	No. 13	764.42	764.40	764.5	762.0	2.4	11.1	0.000088
3B............	No. 2	845.14	845.11	845.3	844.7	0.4	1.9	0.000015
	No. 5	848.90	848.89	849.5	848.0	0.9	4.2	0.000033
	No. 6	803.31	803.31	803.5	802.4	0.9	4.2	0.000033
3C............	No. 2	859.31	859.30	859.9	851.5	7.8	36.0	0.000284
	No. 4	812.38	812.37	812.8	811.4	1.0	4.6	0.000036
	No. 8	776.15	776.12	776.3	774.8	1.4	6.5	0.000051
4B............	No. 4	843.51	845.49	846.1	837.1	6.4	29.6	0.000233
	No. 6	852.83	854.13	854.7	848.1	4.7	21.8	0.000171
	No. 10	817.85	818.97	819.6	812.1	5.7	26.4	0.000208
4C............	No. 1	600.32	600.30	600.9	599.7	0.6	2.8	0.000022
	No. 2	540.88	540.87	541.1	539.1	1.8	8.4	0.000066
	No. 9	515.91	515.87	516.5	514.9	1.0	4.6	0.000036
5B............	No. 1	919.54	922.00	941.6	826.0[a]	93.5	433.0	0.003330
	No. 2	898.10	900.11	918.8	817.2[a]	70.9	328.0	0.002520
	No. 14	903.98	906.07	923.1	815.2[a]	87.8	406.0	0.003160

[a] Were stripped by sandblasting.

TABLE IV.—APPEARANCE OF CORROSION-RESISTANT STEELS AFTER 15 YEARS' OUTDOOR EXPOSURE.

Alloy	Appearance
1A.........	Very slight discoloration. Mostly dirt.
1B.........	80 per cent mottled dark stains. Numerous small rust spots.
1D[a]........	25 per cent dark stain. Few rust spots.
2A.........	30 per cent dark stain. Many very small rust spots.
3A.........	70 per cent dark stain. Some small rust stains.
3B.........	Two panels about 50 per cent mottled dark rust stains. One panel 100 per cent dark stain. Some small rust stains.
3C.........	95 per cent very dark stain. 25 per cent dark red rust.
4B[a]........	80 per cent mottled dark stain. Numerous small rust spots.
4C.........	90 per cent mottled dark stain. Numerous small rust spots.
5B[a]........	100 per cent very dark brown rust. Surface stippled.

[a] Side exposed to east about 50 per cent less stain.

DISCUSSION AND RESULTS

None of the alloys tested showed any serious loss in physical properties over the 15-yr. period of exposure. Alloy 5B is the only one which showed a consistent but minor loss in physical properties over the complete series of tests. Also it is the only steel which had a heavy rust deposit all over the surface and an appreciable weight loss. This alloy is not commonly listed as a corrosion-resistant steel and was included in this test to observe the comparative effect of chromium and nickel on the corrosion resistance of steels.

It is interesting to note that one of the best alloys as far as corrosion resistance is concerned is the 17 per cent chromium alloy, while the other is a standard 18 per cent chromium, 8 per cent nickel alloy of the type 302 composition. The 13 per cent chromium alloys stood up very well as far as loss of physical properties is concerned, although they were completely covered with corrosion at the completion of the test. It will be noted that there is a large variation in carbon content of these 13 per cent chromium steels, which is reflected in the initial physical properties, but which does not seem to have had much effect on the corrosion resistance. None of the specimens used in this test was given the nitric acid passivating treatment which is generally recommended for corrosion-resistant steels. Such a treatment might have had some effect on tarnish resistance of the 18 per cent chromium, 8 per cent nickel type alloys, but since the loss in tensile strength and weight loss on these specimens was negligible, it does not seem that its omission could have affected these results.

CONCLUSIONS

The data obtained from this investigation would indicate that any of the steels tested, with the possible exception of alloy 5B, are satisfactory for use under normal outdoor atmospheric conditions for those applications where appearance is not a requirement. The 17 per cent chromium steel and two of the 18 per cent chromium, 8 per cent nickel alloys stood up very well and seem satisfactory for exposure for applications where appearance is an important factor.

FATIGUE TESTS ON SOME ADDITIONAL COPPER ALLOYS*

By Alton R. Anderson,[1] Emery F. Swan,[2] and Earl W. Palmer[2]

Synopsis

A previous paper (1)[3] from this laboratory presented correlated endurance and tension test results for a number of hard-drawn copper-base alloys. The present paper includes similar data on several other commercially important copper-base alloys, and also data on some of the original alloys in the annealed condition or in additional tempers. Pertinent information regarding composition, fabrication, structure, and other properties is also included.

Small ready-to-finish grain size in a hard-drawn copper-silicon alloy (Everdur 1010) and small final grain size in annealed phosphor bronzes (grades C and D) are shown to result in materials having decidedly better endurance properties than the same alloys finished from or annealed to larger grain sizes.

The data presented here and in the previous paper indicate that for phosphor bronzes the optimum amount of cold working for best endurance behavior decreases with increase in tin content.

Since the publication in 1941 of a paper from this laboratory, "Fatigue Tests on Some Copper Alloys" (**1**), important compilations including newer data have been published by Battelle Memorial Institute (**2**), Everhart, Lindlief, Kanegis, Weissler, and Siegel (**3**), and by Donaldson (**4**). These taken with Gillett's 1932 summary (**5**), give a fairly good coverage of endurance studies on copper and its alloys up to 1943. Since then Burghoff and Blank (**6**) have described the endurance properties of a number of important copper alloys in wire form.

The present paper contains correlated endurance and tension test data on several commercially important copper-base alloys, including some of the previously reported cold-worked alloys now studied in the annealed condition or in additional tempers. Specifically, test data are presented for the following:

Electrolytic tough pitch copper—annealed
63-37 brass—half hard; extra hard
Three leaded brasses—hard
Five phosphor bronzes—several tempers and anneals
70-30 cupro-nickel—hard
Copper-nickel-zinc alloy (Ambrac 850)—hard
18 per cent nickel silver—hard
Two copper-cadmium alloys (Hitenso BB and C)—hard; annealed
Three aluminum bronzes—hard
Two copper-silicon alloys (Everdur 1010 and 1015)—hard; annealed
Copper-aluminum-silicon alloy (Everdur 1014)—hard; annealed

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] Research and Development Engineer, American Metal Hose Branch (formerly Research Assistant, Copper Alloys Research Laboratory), American Brass Co., Waterbury, Conn.

[2] Research Assistant and Research Metallurgist, respectively, Copper Alloys Research Laboratory, American Brass Co., Waterbury, Conn.

[3] The boldface numbers in parentheses refer to the references appended to this paper, see p. 690.

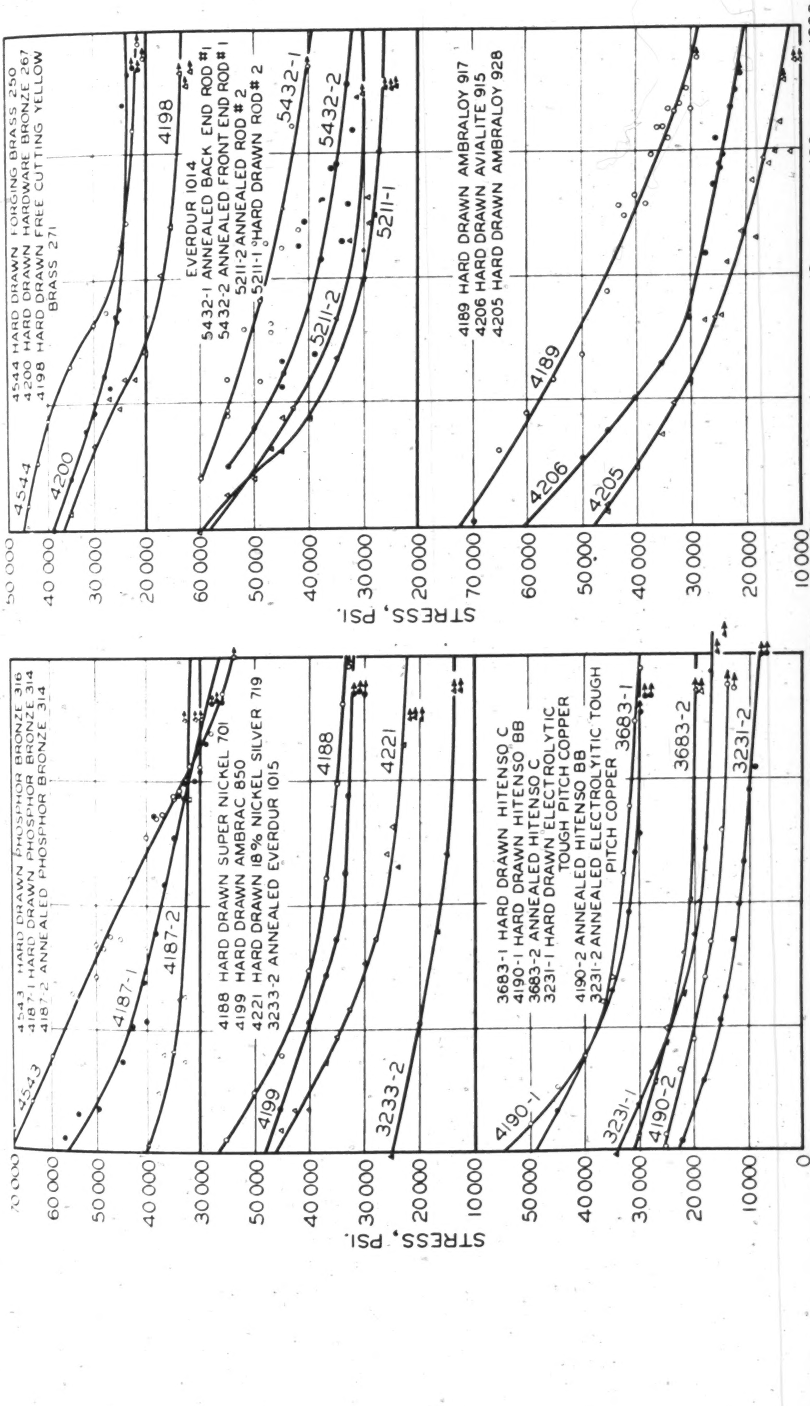

FIG. 1.—*S-N* Curves of Alloys Tested, Showing Experimental Points. Arrows indicate unbroken specimens.

TABLE I.

Tension specimens 0.313 in. in diameter, fatigue specimens 0.300 in. in minimum diameter; machined from 0.500-in. diameter rod. All fatigue specimens machine polished and finished with Alundum 600 and tested at 3500 rpm.

Alloy and Curve Number	Material	Analysis, per cent								
		Cu	Sn	Zn	Mn	Ni	Si	Al	Fe	Other Elements
3231-1	Tough pitch copper (N.E.C.)	99.948[a]								O, 0.031 S, 0.0029
3231-2	Tough pitch copper (N.E.C.)	99.948[a]								O, 0.031 S, 0.0029
4190-1	Hitenso BB	98.88								Cd, 1.12
4190-2	Hitenso BB	98.98								Cd, 1.02
3683-1	Hitenso C	98.67	0.52							Cd, 0.81
3683-2	Hitenso C	98.64	0.60		0.012		0.005			Cd, 0.75; P, 0.004
4200	Hardware bronze 267	85.16		13.00					0.02	Pb, 1.82
4198	Free cutting yellow brass 271	62.26		34.48					0.01	Pb, 3.25
4544	Forging brass 250	58.89		39.28					0.01	Pb, 1.82
4187-1	Phosphor bronze 314	95.20	3.72	0.0	0.21				0.79	P, 0.08
4187-2	Phosphor bronze 314	95.10	3.67	0.12	0.22				0.79	P, 0.08
4543	Phosphor bronze 316	88.35	9.31	0.0	0.78				1.36	P, 0.20
4188	Super Nickel 701	68.96		0.20	0.83	30.01				
4221	Nickel silver 18 per cent 719	64.63		16.22	0.21	18.90			0.03	
4199	Ambrac 850	73.35		6.09	0.47	20.09				
4205	Ambraloy 928	91.96		0.00		0.00	0.012	7.98	0.05	
4206[d]	Avialite 915	89.71	0.38	0.0		0.51	0.018	8.74	0.64	
4189[d]	Ambraloy 917	81.86		0.30	0.94	4.93		9.50	2.47	
5211-1[d]	Everdur 1014	90.78					2.20	6.97	0.05	
5211-2[d]	Everdur 1014	90.78					2.20	6.97	0.05	
5432-1[d]	Everdur 1014	90.98		0.0			2.07	6.89	0.06	
5432-2[d]	Everdur 1014	90.89		0.0			2.05	7.01	0.05	
3233-2	Everdur 1015	98.17		0.00	0.21		1.41		0.06	

Alloy and Curve Number	Material	Commercial Temper Designation	Final Draw (Reduction of Area) or Anneal	Estimated Ready-to-Finish or Final Grain Size, mm.	Endurance Strength at 300,000,000 cycles, psi.	Modulus of Elasticity, psi. (× 10⁻⁶)	Stress, psi.				Yield Strength (0.5 per cent Extension), psi.	Tensile Strength	Elongation, per cent		Reduction of Area, per cent	Rockwell Hardness	Ratios	
							0.0002 per cent Offset	0.002 per cent Offset	0.02 per cent Offset	0.2 per cent Offset			2-in. Gage Length	Gage Length, 4 Times Diameter			Endurance Strength at 300 million / Tensile Strength	Endurance Strength at 300 million / Yield Strength (0.5 per cent Extension)
3231-1	Tough pitch copper (N.E.C.)	Hard	36.0	0.040	17 000	17.77	4 500	8 400	22 500	46 500	47 600	48 800	9.8	14.0	61.1	B47	0.35	0.36
3231-2	Tough pitch copper (N.E.C.)	Annealed	1¼ hr., 600 C.	0.040	9 000	17.80	1 400	2 400	3 400	4 800	5 900	31 100	50.5	57.6	73.6	F43 ± 4	0.29	1.53
4190-1	Hitenso BB	Hard	29.1	0.015	30 000	20.04[b]	11 200[b]	18 700[b]	38 500	70 000	68 700	73 100	8.7	13.6	61.9	B73	0.41	0.44
4190-2	Hitenso BB	Annealed	1 hr., 650 C.	0.050 to 0.055[c]	14 500	18.35	5 100	6 200	7 300	6 800	7 300	35 200	55.8	64.8	81.3	F46	0.41	1.99
3683-1	Hitenso C	Trolley Wire	67.4	0.040	31 000	19.75[b]	14 500[b]	22 600[b]	44 500	73 800	71 300	76 700	9.3	14.1	65.0	B73	0.40	0.43
3683-2	Hitenso C	Annealed	1 hr., 650 C.	0.015 to 0.020[c]	20 000	18.53	8 500	11 600	12 500	12 800	12 800	39 800	53.2	60.8	81.7	F62	0.50	1.56
4200	Hardware bronze 267	Hard	30.0	0.035	23 500	16.99[b]	9 000[b]	15 200[b]	28 000	47 200	48 100	54 900	12.5	20.8	58.5	B68	0.43	0.49
4198	Free cutting yellow brass 271	Hard	21.0	0.070	13 500	11.95[b]	12 700[b]	19 000[b]	30 000	44 300	43 700	52 800	22.8	28.8	47.0	B73	0.26	0.31
4544	Forging brass 250	Hard	21.0	0.015	22 000	14.87[b]	20 500[b]	35 200[b]	48 500	63 800	60 100	75 500	14.5	18.4	38.5	B81	0.29	0.37
4187-1	Phosphor bronze 314	Hard	36.0	0.018	29 000	18.08[b]	23 500[b]	37 000[b]	57 000	78 400	72 300	80 200	10.3	16.6	68.4	B85	0.36	0.40
4187-2	Phosphor bronze 314	Annealed	1 hr., 625 C.	0.008 to 0.010[c]	23 000	17.85[b]	20 500[b]	32 000[b]	25 100	25 600	25 600	54 500	45.0	52.0	76.0	B50	0.59	1.25
4543	Phosphor Bronze 316	Hard	36.0	0.020	28 000	14.41[b]	39 500[b]	....	88 000	111 000	71 300	115 400	6.7	9.6	38.2	B101 ± 4 G87 ± 3	0.24	0.39
4188	Super Nickel 701	Hard	21.0	0.025	34 000	22.85[b]	16 700[b]	26 500[b]	56 000	85 800	84 000	86 900	10.5	16.4	75.4	B87	0.39	0.41
4221	Nickel Silver 18 per cent 719	Hard	21.0	0.030	23 000	19.27[b]	7 500[b]	15 300[b]	33 500	65 000	63 300	72 900	11.8	15.2	60.5	B83	0.32	0.36
4199	Ambrac 850	Hard	21.0	0.045	32 500	21.38[b]	17 000[b]	29 200[b]	52 500	75 700	75 100	76 600	10.0	15.2	71.7	B80	0.42	0.43
4205	Ambraloy 928	Hard	11.5	0.100	14 000	17.30[b]	7 000[b]	11 700[b]	20 500	38 800	40 000	68 400	57.5	64.8	71.3	B77	0.21	0.35
4206[d]	Avialite 915	Hard	11.5	...	22 000	13.27[b]	9 500[b]	17 000[b]	32 500	63 300	53 100	97 500	14.5	18.0	41.3	B97	0.23	0.41
4189[d]	Ambraloy 917	Hard	11.5	...	32 000	16.19[b]	11 500[b]	18 300[b]	37 500	83 500	66 400	124 800	14.2	17.0	21.9	G88 B103	0.26	0.48
5211-1[d]	Everdur 1014	Hard	11.5	...	26 000	16.19[b]	16 800[b]	24 700[b]	42 500	76 000	64 700	107 500	10.3	13.9	22.9	B98	0.24	0.40
5211-2[d]	Everdur 1014	Annealed	1 hr., 600 C.	...	30 000	16.03[b]	16 300[b]	22 600[b]	31 800	42 600	42 800	94 100	32.9	36.8	33.2	B85	0.32	0.70
5432-1[d]	Everdur 1014	Annealed, back end	½ hr., 650 C.	...	41 000	16.76[b]	23 000[b]	33 500[b]	39 600	43 100	43 300	89 900	35.5	41.6	50.3	B84	0.46	0.95
5432-2[d]	Everdur 1014	Annealed, front end	½ hr., 650 C.	...	33 000	16.60[b]	23 000[b]	30 000[b]	35 100	40 400	41 200	87 100	36.3	41.2	45.8	B84	0.38	0.80
3233-2	Everdur 1015	Annealed	1 hr., 650 C.	0.045	14 000	19.82[b]	6 700[b]	7 800[b]	8 600	9 400	10 500	41 600	47.2	55.3	87.2	F54	0.34	1.33

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[a] Copper figure includes silver.

[b] Obtained from deviation curves corrected for true stress and true strain.

[c] Two values indicate range of a nonuniform grain size; single values are for uniform grain size.

[d] Alloys containing two or more phases in which grain size is of little significance.

The present determinations were made on specimens machined from $\frac{1}{2}$-in. diameter commercially fabricated rods, the tension specimens having a 2-in. gage length of 0.313-in. diameter, and the R. R. Moore type rotating-beam fatigue specimens a 0.300-in. minimum diameter. Tension tests were performed in accordance with the refined technique described by Smith (**7**), true stress-true strain corrections (**8**) being applied where significant. Endurance tests were performed on carefully polished specimens (finished with alundum 600) according to the procedure described in the previous paper (**1**). For all tests the specimens were run at 3500 rpm. and each curve carried out at least to 300,000,000 cycles. The figures obtained are believed to approximate ideal endurance strengths in air, from which the effects of various factors such as notches, corrosion, and the like may later be evaluated.

Discussion of the work may conveniently be divided into four parts as follows:

1. Tests on various new materials and on additional tempers of materials previously reported.
2. Tests showing the effects of differences in ready-to-finish grain size on the endurance and other properties of a commercially hard copper alloy (Everdur 1010).
3. Tests showing the effects of differences in grain size on the endurance and other properties of some annealed copper alloys (phosphor bronzes).
4. Tests showing the effects of variations in the amount of cold work on the endurance and other properties of several copper alloys (phosphor bronzes, 63-37 brass and Everdur 1010).

New Materials and Additional Tempers

The stress-cycles curves for the various new materials and for additional tempers of materials previously reported, are plotted in Fig. 1 and data concerning other properties are summarized in Table I. With the aid of this table, it is thought the curves need little explanation. The generally higher endurance-tensile strength ratios of annealed materials, as compared with the same alloys cold-worked, has often been reported (**9, 10, 11, 12, 13, 14**). An unusual situation wherein a metal when cold-worked has a higher endurance-tensile strength ratio than when annealed is indicated by the tests on electrolytic tough pitch copper. (The curve shown for the hard-drawn material is reproduced from the previous paper.) This is in agreement with McAdam's work (**12, 14**) on the same material.

With respect to the data on the copper-aluminum-silicon alloy (Everdur 1014), curves 1 and 2 for alloy 5432 represent the back and front ends, respectively, of the same extruded rod, unmachined specimens from both ends being annealed together under controlled conditions in the same laboratory furnace. The anneals, therefore, are believed to be comparable. Curves 1 and 2 of alloy 5211 represent tests made on hard and annealed specimens, respectively, cut from adjacent segments of a different rod which had been extruded at a somewhat higher temperature.[4] It should also be noted that the anneal used on these latter specimens (5211-2) differed from that used on the first ones. There is no reason to believe that these four series of tests include the extremes in endurance performance obtainable with material of this general composition. However the alloy obviously is subject to considerable variation in properties with mill treatment.

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[4] With this alloy, extrusion temperature has a profound effect on the structure of the metal, over and beyond grain size and amount of "cold" work. Material extruded relatively hot leaves the die as one phase (kappa) and thereafter precipitates out the second phase (alpha). At lower temperatures the material is in a duplex condition during extrusion.

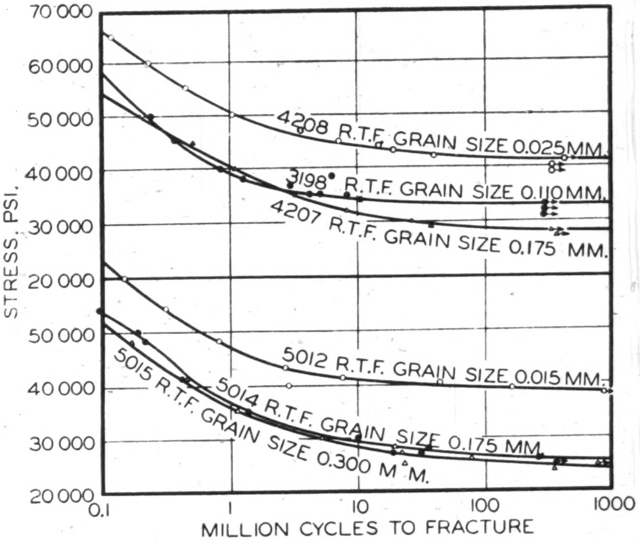

FIG. 2.–*S-N* Curves of Hard-Drawn Everdur 1010 Showing Effect of Ready-to-Finish Grain Size on Endurance.

TABLE II. COMPARISON OF COMMERCIALLY HARD[a] EVERDUR 1010 ROD FINISHED FROM DIFFERENT GRAIN SIZES.

Grain size prior to final cold work, mm. Laboratory alloy	0.175 No. 4207	0.110 No. 3198	0.025 No. 4208	0.300 No. 5015	0.175 No. 5014	0.015 No. 5012
Analysis, per cent						
Copper	95.65	95.51	95.68	95.65	95.65	95.65
Silicon	3.07	3.11	3.08	3.20	3.20	3.20
Manganese	0.92	1.04	0.94	0.94	0.94	0.94
Zinc	0.20	0.15	0.15	0.20	0.20	0.20
Iron	0.10	0.15	0.11	0.13	0.13	0.13
Modulus of elasticity, psi. ($\times 10^{-6}$)[b]	14.20	14.65	14.47	14.58	14.41	14.44
Stress for 0.0002% offset, psi.[b,c]	16 100	19 300	19 500	8 000	12 400	10 000
Stress for 0.002% offset, psi.[b]	25 600	28 500	30 400	18 700	20 100	17 300
Stress for 0.02% offset, psi.	47 500	47 700	53 000	40 700	41 700	42 000
Stress for 0.2% offset, psi.	82 800	....	92 000	76 800	77 800	84 000
Yield strength, 0.5% Ext., psi.	63 600	64 200	66 100	61 200	61 100	62 300
Tensile strength, psi	95 000	97 100	106 900	93 500	95 300	107 100
Elongation, per cent						
Gage length = 2 × 0.313 in. diameter	11.7	12.8	8.8	14.5	13.3	11.0
Gage length = 4D × 0.313 in. diameter	17.2	18.4	12.8	21.6	19.2	18.4
Reduction of area, per cent	55.8	61.7	39.0	71.3	66.5	58.4
Rockwell hardness	B97.0	B99	B100	B96 G77	B97 G77	B98 G80
Endurance Strengths, psi.[d]						
for 0.1×10^6 cycles	55 000	59 000	66 500	52 000	53 500	63 500
for 1×10^6 cycles	41 000	40 000	51 000	36 000	37 000	47 000
for 10×10^6 cycles	31 500	34 500	44 000	28 500	29 500	41 000
for 100×10^6 cycles	28 500	33 500	42 000	25 500	26 500	39 500
for 300×10^6 cycles	28 000	33 500	41 500	24 500	26 000	39 000
for 1000×10^6 cycles	28 000	33 500	41 000	24 000	25 500	38 500
estimated uncertainties	±1000	±1000	±1000	±1000	±1000	±1000
Ratios						
$\frac{\text{End. Str. at } 300 \times 10^6 \text{ cycles}^e}{\text{Tensile Strength}}$	0.30	0.35	0.39	0.26	0.27	0.36
$\frac{\text{End. Str. at } 300 \times 10^6 \text{ cycles}}{\text{Yield Strength (0.5\% Extension)}}$	0.44	0.52	0.63	0.40	0.43	0.63

[a] Final cold reduction, 39 per cent.
[b] Values corrected for true stress and true strain.
[c] Sometimes called "proportional limit, deviation of 0.0002 per cent."
[d] As read from *S-N* curves, values rounded off to nearest 500 psi.
[e] Frequently called "endurance ratio."

and under some conditions may show inconsistent behavior under repeated stress (see curves 5432-1 and -2). However, it should be noted that the 41,000-psi. endurance strength at 300,000,000 cycles obtained for alloy 5432-1 (back end of push) is high for a copper-base alloy.

As will be observed, many of the tests were continued well beyond the arbitrary 300,000,000 cycle point, frequently assumed to be sufficient to establish the endurance *limit* of copper-base alloys. Only a few of the curves reported in this paper appear to have become asymptotic at this point and many are far from horizontal even at 1,000,000,000 cycles. If true endurance limits exist for such materials, they apparently are reached only after astronomical numbers of stress cycles. In predicting the behavior of such alloys, caution should be observed in extrapolating beyond the point actually reached by the tests.

EFFECT OF READY-TO-FINISH GRAIN SIZE IN A HARD-DRAWN COPPER ALLOY—(EVERDUR 1010)

The tests on hard-drawn Everdur 1010 with different ready-to-finish grain sizes (*S-N* curves shown in Fig. 2 and properties given in Table II) actually represent two independent studies of this factor. Alloys 4208, 3198, and 4207

TABLE III.—COMPARISON OF DIFFERENT TEMPERS OF ROD—PHOSPHOR BRONZE GRADE A.

Copper, per cent	95.27
Tin, per cent	4.32
Phosphorus, per cent	0.38
Lead, per cent	0.003
Iron, per cent	0.025

Temper	Annealed at 550 C.	Half Hard	Hard	Extra hard
Alloy and curve number	3024–2	3023	3024–1	3025
Grain size, mm	0.025	0.070 RTF[e]	0.090 RTF[e]	0.065 RTF[e]
Final draw, reduction of area, per cent	0	15.2	30.1	50.1
Modulus of elasticity, psi. ($\times 10^{-6}$)[a]	17.37	17.34	16.38	14.91
Stress for 0.0002% offset, psi.[a, b]	13 400	30 000	35 600	34 500
Stress for 0.002% offset, psi.[a]	19 900	34 000	39 500	43 800
Stress for 0.02% offset, psi.	21 200	41 700	48 500	59 300
Stress for 0.2% offset, psi.	20 200	49 700	61 700	84 000
Yield strength, 0.5% Ext., psi.	20 200	49 600	60 000	67 600
Tensile strength, psi.	50 600	56 700	69 800	96 600
Elongation, per cent				
Gage length = 2 × 0.313 in. diameter	64.2	45.8	22.7	11.2
Gage length = 4D × 0.313 in. diameter	73.8	54.9	32.5	17.1
Reduction of area, per cent	82.9	82.6	78.7	71.3
Rockwell hardness	B34	B72	B92	B92
Endurance strength, psi.[c]				
for 0.1 × 10^6 cycles	39 000	43 200	51 000	56 000
for 1 × 10^6 cycles	32 500	34 000	36 500	43 000
for 10 × 10^6 cycles	30 000	29 500	31 000	35 000
for 100 × 10^6 cycles	29 000	28 000	30 000	33 000
for 300 × 10^6 cycles	29 000	27 500	29 000	32 000
for 1000 × 10^6 cycles	29 000	27 500	28 500	32 000
estimated uncertainty	±1000	±2000	±2000	±3000
Ratios				
$\frac{\text{End. Str. at } 300 \times 10^6 \text{ cycles}^d}{\text{Tensile Strength}}$	0.57	0.49	0.41	0.33
$\frac{\text{End. Str. at } 300 \times 10^6 \text{ cycles}}{\text{Yield Strength (0.5\% Extension)}}$	1.44	0.55	0.48	0.47

[a] Values corrected for true stress and true strain.
[b] Sometimes called "proportional limit, deviation of 0.0002 per cent."
[c] As read from *S-N* curves, values rounded off to nearest 500 psi.
[d] Frequently called "endurance ratio."
[e] Ready-to-finish.

were fabricated in the mill without laboratory supervision or observation, and also vary from one another somewhat in composition. After these tests had been completed, it was considered desirable to run a second series of tests on new material to determine whether the very significant differences in endurance properties observed were actually related to grain size, or might be attributable to variations in composition or treatment of the material. Hence, the new material (alloys 5012, 5014, and 5015) was fabricated from a single heat under the supervision of one of the authors, and it is believed that variables other than those resulting from intentional differences in temperatures of the anneal prior to the final cold-working are negligible. The results on this second series showed a similar variation of endurance properties with ready-to-finish grain size, indicating clearly that this factor (or some other associated with it, possibly directionality) is significant. The superiority in endurance of rods finished from a small grain size appears to be of considerable importance for this material. While strictly comparable data are not available for other compositions, there is good reason to believe that the effect of ready-to-finish grain size is of general significance in hard-drawn copper-base alloys. The direction of the effect of grain size on the fatigue properties of metals has been known for some time, as indicated by Rawdon (**15**), Greenall and Gohn (**11**) and Burghoff (**16**). In this country (**17**) this factor has generally been considered of minor importance; Seelig (**18**) indicates that in Europe greater weight is attached to it.

EFFECT OF GRAIN SIZE IN ANNEALED PHOSPHOR BRONZES

Results of endurance tests on annealed specimens of several phosphor bronzes are shown in Fig. 3 and their properties are included in Tables III, IV, and V. A number of points are thought to deserve consideration. The differences between endurance strengths of the fine-grained materials and those having a coarser grain are decidedly significant. The improvement in nominal endurance strength at 300,000,000 cycles for the fine-grained material as compared with the coarse, for either grade C or grade D phosphor bronze, is approximately 10,000 psi., which represents an increase of about 40 per cent. In general the

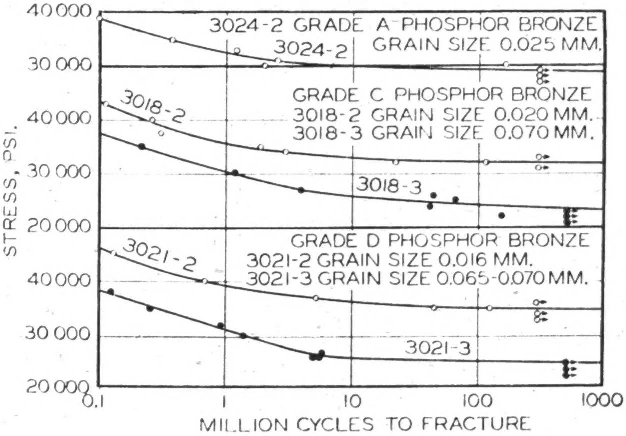

FIG. 3.—*S-N* Curves of Annealed Phosphor Bronzes Showing Effect of Grain Size on Endurance.

observed increases in endurance strengths are considerably greater than the corresponding increases in tensile properties. The endurance-tensile strength ratios for the fine-grained annealed phosphor bronzes are unusually high for copper-base alloys, and the endurance-yield strength (0.5 per cent extension) ratios for all five annealed phosphor bronzes reported are above unity. This latter characteristic was pointed out by McAdam (**14**) some time ago, and is thought to indicate either that the nominal (computed) endurance stresses are greater than the true stresses, because of a nonlinear stress-strain relationship, or that during the endurance test the tensile properties of the surface layers of the specimen are raised, or both.

Despite an endurance-yield strength ratio above unity, the use of these copper alloys for endurance applications at stresses above their yield strengths certainly cannot be recommended. Where appreciable overstresses are not anticipated, it would seem safe to use the yield strength of these annealed phosphor bronzes as the endurance design stress (subject, of course, to the usual factor of safety), even where this yield strength is above the endurance strength of the cold-worked alloy.

EFFECTS OF COLD WORK

The effects of cold work in raising the

TABLE IV.—COMPARISON OF DIFFERENT TEMPERS OF ROD—PHOSPHOR BRONZE GRADE C.

Copper, per cent	91.78
Tin, per cent	8.10
Phosphorus, per cent	0.08
Lead, per cent	0.002
Iron, per cent	0.043

Temper	Annealed at 550 C.	Annealed at 625 C.	Half Hard	Hard	Extra Hard
Alloy and curve number	3018–2	3018–3	3017	3018–1	3019
Grain size, mm	0.020	0.070	0.100 RTF[e]	0.090 to 0.100 RTF[e]	0.110 RTF[e]
Final draw, reduction of area, per cent	0	0	15.2	30.1	50.1
Modulus of elasticity, psi. ($\times 10^{-6}$)[a]	16.77	16.72	16.43	15.00	13.80
Stress for 0.0002% offset, psi.[a, b]	21 000	17 000	30 000	36 000	34 300
Stress for 0.002% offset, psi.[a]	24 300	19 700	33 000	39 700	43 600
Stress for 0.02% offset, psi.	24 900	20 900	39 300	48 500	61 500
Stress for 0.2% offset, psi.	24 500	20 000	49 100	64 500	93 100
Yield strength, 0.5 per cent Ext., psi.	24 600	20 200	49 100	60 200	65 300
Tensile strength, psi	59 200	55 500	66 000	81 000	110 300
Elongation, per cent					
Gage length = 2 × 0.313 in. diameter	78.3	78.8	47.8	26.7	10.0
Gage length = 4D × 0.313 in. diameter	83.6	86.4	56.5	35.2	14.7
Reduction of area, per cent	81.2	84.0	81.1	77.3	69.2
Rockwell hardness	B50	B41	B80	B97	B98
Endurance strengths, psi.[c]					
for 0.1×10^6 cycles	43 000	37 500	50 000	47 000	58 000
for 1×10^6 cycles	35 500	30 500	38 000	40 000	48 000
for 10×10^6 cycles	33 000	26 000	31 000	37 000	38 000
for 100×10^6 cycles	32 000	24 000	30 000	35 000	31 000
for 300×10^6 cycles	32 000	23 500	29 000	35 000	29 000
for 1000×10^6 cycles	32 000	23 000	29 000	34 000	29 000
estimated uncertainty	±1000	±1 500	±2000	±3000	±5000
Ratios					
$\frac{\text{End. Str. at } 300 \times 10^{6} \text{ cycles}}{\text{Tensile Strength}}$[d]	0.54	0.42	0.44	0.43	0.26
$\frac{\text{End. Str. at } 300 \times 10^{6} \text{ cycles}}{\text{Yield Strength (0.5\% Extension)}}$	1.30	1.16	0.59	0.58	0.44

[a] Values corrected for true stress and true strain.

[b] Sometimes called "proportional limit, deviation of 0.0002 per cent."

[c] As read from *S-N* curves, values rounded off to nearest 1000 psi. for half hard, hard, and extra hard rods, to nearest 500 psi. for annealed rods.

[d] Frequently called "endurance ratio."

[e] Ready-to-finish.

endurance strengths of metals is well known and has received considerable attention. While this phenomenon is generally accepted, it is known that excessive amounts of cold working may have an adverse effect. In view of the pronounced grain-size effect here reported for some alloys, it is felt that any study of the effects of cold working must take into consideration the ready-to-finish grain sizes of the materials compared. With this in mind, we felt it desirable that data presented in the previous paper for four tempers of Everdur 1010 should be re-examined. The pertinent information is tabulated below:

Alloy	Temper	Anneal or Final Reduction of Area, per cent	Grain Size, mm.	Endurance Strength at 300 000 000 cycles, psi.
3198-A....	Soft	0.5 hr. at 700 C.	0.085	18 800[a]
3410.......	Quarter-hard	8.0	0.035 Ready-to-finish	31 500[b]
3411.......	Half-hard	20.0	0.065 Ready-to-finish	30 500[b]
3198.......	Hard	39.0	0.125 Ready-to-finish	33 600[a]

[a] Tests run at 3500 rpm. on 0.300-in. minimum diameter specimens.

[b] Tests run at 1600 rpm. on 0.250-in. minimum diameter specimens.

TABLE V. COMPARISON OF DIFFERENT TEMPERS OF ROD—PHOSPHOR BRONZE GRADE D.

Copper, per cent	89.86
Tin, per cent	9.76
Phosphorus, per cent	0.12
Zinc, per cent	0.20
Lead, per cent	0.00
Iron, per cent	0.06

Temper	Annealed at 550 C.	Annealed at 650 C.	Half Hard	Hard	Extra Hard
Alloy and curve number	3021–2	3021–3	3020	3021–1	3022
Grain size, mm.	0.016	0.065 to 0.070	0.075 RTF[e]	0.090 RTF[e]	0.090 to 0.100 RTF[e]
Final draw, reduction of area, per cent	0	0	15.2	30.1	50.1
Modulus of elasticity, psi. (× 10^{-6})[a]	16.30	16.20	15.40	14.07	13.22
Stress for 0.0002% offset, psi.[a, b]	24 000	16 000	18 000	32 800	29 300
Stress for 0.002% offset, psi.[a]	27 800	21 200	27 500	35 300	43 500
Stress for 0.02% offset, psi.	29 100	22 300	37 000	50 500	65 500
Stress for 0.2% offset, psi.	28 400	21 200	50 300	70 400	109 000
Yield strength, 0.5% Ext., psi.	28 400	21 300	49 700	60 000	63 500
Tensile strength, psi.	68 000	62 200	76 600	92 100	127 500
Elongation, per cent					
Gage length = 2 × 0.313 in. diameter	70.0	75.3	45.8	23.2	8.5
Gage length = 4D × 0.313 in. diameter	75.7	82.4	53.6	30.4	12.8
Reduction of area, per cent	74.5	78.4	72.0	63.6	56.4
Rockwell hardness	B60	B49	B92	B97	B103 G87
Endurance strengths, psi.[c]					
for 0.1 × 10^6 cycles	46 000	38 000	53 000	55 000	64 500
for 1 × 10^6 cycles	39 500	31 500	37 000	42 000	48 000
for 10 × 10^6 cycles	36 500	25 500	32 000	31 000	34 000
for 100 × 10^6 cycles	35 500	25 000	30 000	25 000	25 000
for 300 × 10^6 cycles	35 000	25 000	30 000	23 000	23 500
for 1000 × 10^6 cycles	35 000	25 000	30 000	21 000	23 000
estimated uncertainty	±1000	±1000	±2000	±2000	±2000
Ratio:					
$\frac{\text{End. Str. at } 300 \times 10^6 \text{ cycles}^d}{\text{Tensile Strength}}$	0.51	0.40	0.39	0.25	0.18
$\frac{\text{End. Str. at } 300 \times 10^6 \text{ cycles}}{\text{Yield Strength (0.5\% Extension)}}$	1.23	1.17	0.60	0.38	0.37

[a] Values corrected for true stress and true strain.

[b] Sometimes called "proportional limit, deviation of 0.0002 per cent."

[c] As read from S-N curves, values rounded off to nearest 1000 psi. for half hard, hard, and extra hard rods, to nearest psi. for annealed rods.

[d] Frequently called "endurance ratio."

[e] Ready-to-finish.

It will be observed that the quarter-hard material was found to have a slightly higher endurance strength than the half-hard metal, but that it was finished from a smaller grain size. Had they both been finished from the same grain size, it is believed that the half-hard material would have had a higher endurance strength than the quarter-hard metal. Similarly, a greater difference between the endurance strengths of the hard and half-hard tempers would be expected had both rods been finished from the same grain size.

For comparative purposes the *S-N* curves for three materials, 63-37 brass, grade A phosphor bronze, and grade D phosphor bronze, each in half-hard, hard, and extra-hard tempers, are plotted together in Fig. 4. Other pertinent data for these materials may be found in Tables III, V, and VI.

The 63-37 brass shows an increase in endurance strength (at 300,000,000 cycles) with cold working which is almost exactly in proportion to its increase in ultimate tensile strength, but the harder tempers were finished from somewhat smaller grain sizes. Were this not the case, the increase in endurance strength would probably not have been so great. It will be observed that the extra-hard temper is much less uniform in fatigue properties, as evidenced by the relatively greater "scatter" of points on the *S-N* diagram.

The grade A phosphor bronze also increases in endurance strength with

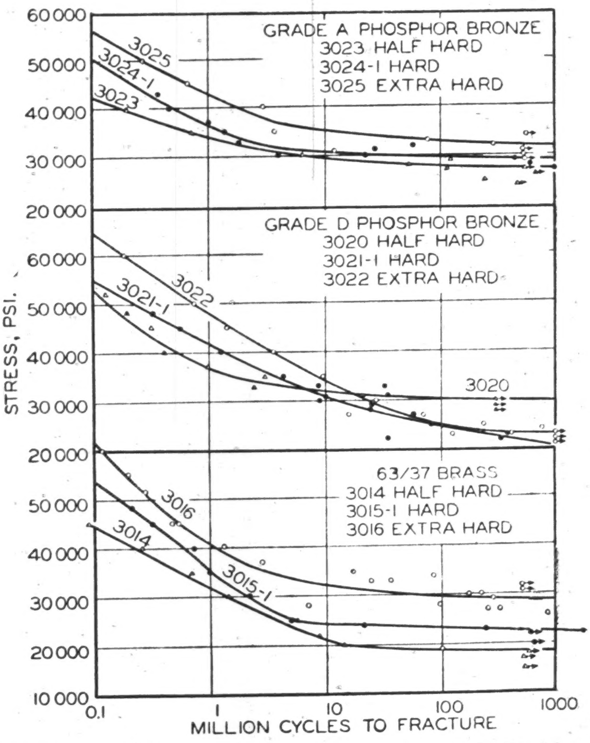

Fig. 4.—*S-N* Curves of Alloys with Varying Amounts of Cold-Working, Showing Effect of Temper on Endurance Strengths.

amount of cold work, but not in proportion with tensile strength. In fact, the endurance-tensile strength ratio steadily decreases. The ready-to-finish grain sizes of the half-hard and extra-hard tempers were reasonably identical; that of the hard material was larger, which may account for the smaller difference between the half-hard and hard tempers than between the hard and extra-hard rods. No significant differences in "scatter" of *S-N* points were observed with the various tempers of this alloy. Attention is called to the endurance strengths of the fine-grained annealed material, which, except for small numbers of cycles, are as great or greater than the corresponding endurance strengths of the half-hard and hard tempers, though not as great as those of the extra-hard material.

The results obtained on grade D phosphor bronze are not in line with those obtained on the other two alloys of this group and on Everdur 1010, in that the endurance strengths (for large numbers of cycles) are lower for the hard and extra-hard tempers than for the half-hard temper. The half-hard material was finished from a 0.075-mm. grain size while the hard and extra-hard materials were finished from 0.090 and 0.100-mm. grain sizes, respectively. It is possible that grain-size differences account for the superiority of the half-hard metal over the hard and the *slight* superiority of the extra-hard over the hard temper. The data, however, are

TABLE VI.—COMPARISON OF DIFFERENT TEMPERS OF 63-37 BRASS ROD.

Copper, per cent	63.00
Zinc, per cent	36.96
Lead, per cent	0.03
Iron, per cent	0.01

Temper	Half Hard	Hard	Extra Hard
Alloy and curve number	3014	3015	3016–1
Alpha grain size, mm	0.070 RTF[e]	0.060 RTF[e]	0.040 to 0.050 RTF[e]
Final draw, reduction of area, per cent	15.2	30.1	50.1
Modulus of elasticity, psi. ($\times 10^{-6}$)[a]	15.64	15.72	15.38
Stress for 0.0002% offset, psi.[a, b]	22 000	17 600	14 000
Stress for 0.002% offset, psi.[a]	28 500	26 000	23 000
Stress for 0.02% offset, psi.	35 700	37 500	38 500
Stress for 0.2% offset, psi.	43 100	55 500	67 000
Yield strength, 0.5% Ext., psi.	43 300	53 900	59 000
Tensile strength, psi.	57 200	72 200	91 600
Elongation, per cent			
Gage length = 2 × 0.313 in. diameter	36.5	17.8	9.8
Gage length = 4D × 0.313 in. diameter	44.0	24.4	14.8
Reduction of area, per cent	70.2	61.6	58.7
Rockwell hardness	B71	B82	B93
Endurance strengths, psi.[c]			
for 0.1×10^6 cycles	44 500	54 000	62 000
for 1×10^6 cycles	33 000	36 000	41 000
for 10×10^6 cycles	21 500	24 000	33 000
for 100×10^6 cycles	19 000	23 000	30 000
for 300×10^6 cycles	18 500	22 500	29 500
for 1000×10^6 cycles	18 000	22 000	29 000
estimated uncertainty	±1000	±1000	±3000
Ratios			
$\frac{\text{End. Str. at } 300 \times 10^6 \text{ cycles}^d}{\text{Tensile Strength}}$	0.32	0.31	0.32
$\frac{\text{End. Str. at } 300 \times 10^6 \text{ cycles}}{\text{Yield Strength (0.5\% Extension)}}$	0.43	0.42	0.50

[a] Values corrected for true stress and true strain.
[b] Sometimes called "proportional limit, deviation of 0.0002 per cent."
[c] As read from *S-N* curves, values rounded off to nearest 500 psi.
[d] Frequently called "endurance ratio."
[e] Ready-to-finish.

inadequate to establish this point with any degree of assurance.

Considering only the amount of cold working, the data presented here and in the previous paper would seem to indicate that for phosphor bronzes the optimum amount of cold working decreases with an increase in tin content. For the 4 per cent tin bronze, the extra-hard temper shows up best; for the 8 per cent tin bronze, the hard temper is highest; for the 10 per cent tin bronze, the half-hard material has the highest 300,000,000 cycles endurance strength. McAdam (**14**) also noted this tendency, but in view of the possible large effects of ready-to-finish grain size, which must be taken into consideration, it is thought deserving of further investigation.

Acknowledgments:

The work presented in this paper was carried out in the Copper Alloys Research Laboratory of The American Brass Co. under the general direction of John R. Freeman, Jr., Technical Manager. The authors wish to express their appreciation to F. H. Wilson and J. J. Breen, who assisted in performing the tension tests herein reported.

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(**18**) R. P. Seelig, "Recent Developments in European Research on Fatigue of Metals," ASTM BULLETIN, No. 94, pp. 23–30; No. 95, pp. 15–22 (1938).

DISCUSSION

Mr. George R. Gohn.[1]—Mr. Palmer has brought forth a very important point, that is, the effect of grain size in the ready-to-finish anneal on the ultimate physical properties and, particularly, the fatigue properties which you may get in non-ferrous materials.

Back in 1937,[2] we presented a small amount of data on nickel silver sheet, which indicated that material running 0.015 to 0.017 mm. in average grain size (ready-to-finish anneal) had a higher endurance limit than material of larger grain size.

We believe that this phenomenon is probably something that is peculiar to copper-base alloys, aluminum and magnesium alloys, and the cold-rolled steels. The steel people apparently are not as likely to encounter it as the non-ferrous investigators because, in most cases, they are dealing with materials whose physical properties are obtained by various heat treatments.

We believe this variation in the ready-to-finish anneal may acount for the variation in fatigue results reported by various investigators. I am very glad that Mr. Palmer has emphasized this point.

Mr. John M. Lessells.[3]—This paper is of interest in regard to the slope of the S-N diagram as affected by the treatment given to the material.

In the case of steel having protected surfaces produced by nitriding or shot peening, fractures are generally obtained at a much higher number of stress cycles than would be experienced with the same material in the absence of surface protection. This raises the question whether by long periods of cyclic stressing of material with protected surfaces the beneficial effects are not finally eliminated. This might be possible in the case of the shot-peened surface.

Gough[4] has indicated that in the fatigue testing of non-ferrous metals and alloys, where similar S-N curves with a drooping characteristic may be experienced, that two values of stress cycles should be recorded; namely, that upon which the endurance limit is based and that at which the S-N curve becomes and remains horizontal. It appears that the treatment which the material has received will determine whether a definite endurance limit is obtained.

It would be of interest to hear some comment from the author as regards his experience with copper alloys.

Mr. E. W. Palmer (*author*).—We quite agree with Mr. Gohn that variation in the ready-to-finish anneal may account for the variation in fatigue results reported by the several investigators of non-ferrous materials.

The work of Greenall and Gohn on nickel-silver sheet is mentioned in the paper, and is possibly the first evidence in the literature that hard material finished from a small grain size has a higher endurance limit than that finished from a large grain size. However, it was known that tensile properties generally were slightly higher for the fine-grained material, and the disproportionately large increase in endurance limit does not appear to have been then appreciated.

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[1] Bell Telephone Labs., Inc., New York, N.Y.

[2] C. H. Greenall and G. R. Gohn, "Fatigue Properties of Non-Ferrous Sheet Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part II, p. 160 (1937).

[3] Associate Professor of Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

[4] In a series of unpublished lectures given at Massachusetts Inst. of Technology, in 1937.

In reply to Mr. Lessells, our experience with copper alloys would indicate that a steadily falling *S-N* curve is characteristic of many two-phase alloys, especially when cold-worked, and of highly alloyed single-phase alloys given relatively large amounts of cold work. The effect of cold work on grade D phosphor bronze is well shown in the middle group of curves of Fig. 4. We suspect that the distribution and intensity of internal micro-stresses may have a great deal to do with determining the slope of the *S-N* curve.

MR. ALBERT I. BLANK.[5]—The authors have presented some very valuable endurance data on copper-base alloys. Several of the endurance strength values included in the paper are especially interesting. In the case of phosphor bronze 314 the endurance strength at 300,000,000 cycles is 29,000 psi. for the hard-drawn temper, whereas that for the annealed temper is 32,000 psi. The case of Everdur 1014 is similar—the hard-drawn temper has an endurance strength of 26,000 psi. and the annealed temper 30,000 psi. Annealed tempers ordinarily have lower endurance properties than drawn tempers. I wonder whether the authors can account for the unusual circumstances pertaining to these two materials which have higher endurance strength in the annealed condition.

MR. PALMER.—With regard to the relative endurance strengths at 300,000,000 cycles of hard and annealed phosphor bronze 314, we have no explanation to offer at present as to why this alloy has the better endurance strength, for large numbers of cycles, when annealed. In the case of Everdur 1014, however, where similar behavior was found, we believe the two-phase structures of the alloy may be responsible. It seems probable that the cold working of such a structure results in high internal micro-stresses, which add to the stresses imposed in the test and result in a lower effective endurance strength. We are planning additional work on stress-relieved materials of this type which may result in some clarification of this problem.

It should be noted that two entirely different types of behavior are represented by these two alloys. The annealed phosphor bronze becomes superior to hard-drawn material of the same composition only where more than 100,000,000 cycles of stress are involved. The annealed Everdur 1014, on the other hand, is superior to its hard-drawn counterpart throughout practically the whole range of stress cycles studied. Only for very low numbers of cycles, below about 500,000, is the hard-drawn material perhaps slightly superior.

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[5] Assistant Research Metallurgist, Chase Brass & Copper Co., Waterbury, Conn.

A STUDY OF THE DAMAGING EFFECT OF FATIGUE STRESSING ON X4130 STEEL*

By J. A. Bennett[1]

Synopsis

The damaging effect of fatigue stressing above the endurance limit was investigated with notched specimens of S.A.E. X4130 steel. The damage was measured by the decrease in endurance at another stress. A deflection method for detecting the formation of a fatigue crack permitted the damage measurement to be limited chiefly to the pre-crack stage. The results showed that the apparent rate of damage depends on the stress history. If the pre-stress is higher than the test stress the damage occurs rapidly at first, then more slowly. The reverse is true if the damaging stress is lower than that used to measure the damage.

Tests also were made with smooth specimens in an effort to determine the cumulative damage caused by fatigue at more than one stress. Two methods were developed for extrapolating to determine the point at which fatigue cracking starts, so the damaging cycle ratios could be based on the life prior to cracking. Complete *S-N* curves were determined for specimens after each of eight different damaging treatments. Using these curves, a method of expressing damage was developed which permitted the direct addition of damage occurring at different stresses. The reliability of this method was checked by testing specimens after fatigue loading at two or more different stresses and comparing the results with predictions based on the addition of the indicated damage. The agreement was within the experimental error.

Most ferrous metals exhibit a fatigue limit such that if a sample of the metal is subjected to a range of stress greater than the fatigue limit, a crack will form which eventually will lead to fracture. Prior to formation of the first crack there is a change taking place in the metal which has been detectable only by means of a fatigue test. For example if a specimen is subjected to an overstress for one half the number of cycles required to cause fracture, the usual mechanical properties of the material, other than fatigue, appear to be unchanged. However, the fatigue properties, both the endurance at stresses above the fatigue limit and the fatigue limit itself, will be lowered. The purpose of the work described in this report was to study some of the phenomena associated with this damaging process, particularly with a view to making the information more readily applicable in the design of steel structures against failure under alternating loads.

Previous investigations of fatigue damage have dealt largely with smooth specimens, although the majority of fatigue failures in service propagate from points of stress concentration. Therefore it was thought worth while to make a thorough study using notched specimens, for comparison with the results with smooth specimens. This study comprised the first part of the present investigation and the results are discussed in the section on Test Results with Notched Specimens of this report. The work was done under the auspices of, and with financial assistance from, the National Advisory Committee for Aeronautics, and has been described in a publication of this Committee (**1**).[2]

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

[1] Division of Metallurgy, National Bureau of Standard, Washington, D. C.

A large proportion of the highly stressed machine elements in service probably are stressed above their fatigue limit at least occasionally during their life. It is important, therefore, to know how much damage is done by this overstressing. Since the rate of damage in a simple test is known (**2**) to be a complicated function of the stress history, it has not been possible to predict systematically the performance of material in service where the stress variation follows a random schedule. The second part of this investigation dealt with the study of the damage caused by combinations of different ranges of stress. It was hoped that part of the gap between the simple laboratory test and the random fluctuation of stress usually encountered under service conditions could be bridged by making the loading schedule more and more complicated. The results of this part of the investigation are described in the section on Test Results with Smooth Specimens of this report.

MATERIALS AND SPECIMENS

All of the fatigue tests included in this report were made on R. R. Moore type machines. The specimens were of normalized S.A.E. X4130 steel, a different heat being used for each part of the investigation. The mechanical properties of the two are listed in Table I.

The notched specimens had a cylindrical test section 0.35 in. in diameter, into which was cut a semicircular circumferential notch 0.05 in. deep and 0.05 in. in radius. The stresses listed for these specimens were calculated for the minimum section of 0.25-in. diameter without regard to stress concentration. The notches were finished with a copper wire, slightly smaller in diameter than the notch, charged with a slurry of No. 302 emery in water. The specimens were rotated slowly in a lathe while the wire, held at right angles to the axis of the specimen, was rotated rapidly. Thus the fine polishing scratches at the bottom of the groove were substantially parallel to the axis of the specimen.

TABLE I.—MECHANICAL PROPERTIES OF THE STEELS.

	M 390	M 357
	Used for Notched Specimens	Used for Smooth Specimens
Yield strength, 0.2 per cent offset, psi	62 500	63 000
Ultimate tensile strength, psi	104 200	98 000
Elongation in 1 in., per cent	27	29
Reduction of area, per cent	57	60
Rockwell hardness, B scale	90	89

The smooth specimens had a minimum section 0.25 in. in diameter, with a contour radius of $9\frac{7}{8}$ in. These were polished with Aloxite polishing paper belts mounted on a rubber drum $5\frac{1}{2}$ in. in diameter. The specimens were mechanically rotated about 15 rpm. while being held lightly against the rotating drum, the axis of the specimen being at right angles to the axis of the drum. The drum speed was 1750 rpm., and the specimens usually could be polished satisfactorily in less than a minute. The only precaution necessary was to avoid heavy pressure of the specimen against the abrasive. This polishing method has proved economical in preparing standard R. R. Moore specimens. While the resulting surface is not highly polished, the results obtained with these specimens are as consistent as those with a higher polish. All of the specimens of both the notched and smooth types were examined with a low-power microscope to make sure that none with circumferential scratches was tested.

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[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 711.

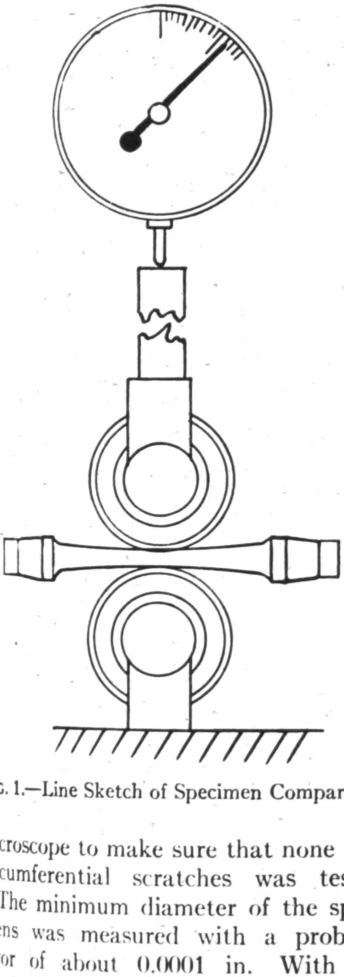

FIG. 1.—Line Sketch of Specimen Comparator.

The minimum diameter of the specimens was measured with a probable error of about 0.0001 in. With the notched specimens this was done with the aid of calibrated gage wires slightly less than 0.1 in. in diameter placed in the groove, the distance between the outsides of the wires being measured with micrometer calipers. The smooth specimens were measured with a vertical comparator using a 0.0001-in. dial gage. A line sketch of this instrument is shown in Fig. 1. In place of the usual anvils, contact with the specimen was made with two ball bearings about $1\frac{5}{8}$ in. in outside diameter. The specimen was placed between the bearings with the axis of the specimen at right angles to those of the bearings. In this way the specimen could be moved back and forth parallel to its length to locate the minimum section without danger of scratching the surface.

For some of the work with smooth specimens it was necessary to have a reference mark at the minimum section of the specimens. To locate this, a light source was set up so that an edge of the projected beam coincided with the line between the centers of the comparator bearings. Thus when a minimum reading was obtained on the dial, the edge of the beam passed across the minimum section of the specimen. A reference mark was made at this position with a very small drop of rubber cement placed on the specimen with a fine wire.

TESTING MACHINES AND SPECIAL APPARATUS

All of the R. R. Moore type machines used in this investigation were equipped with ball bearing spindles and all tests were run at 3600 rpm. A variable autotransformer was used to reduce the speed of the 10,000-rpm. motors with which some of the machines were equipped.

Certain auxiliary apparatus was used in this investigation, as follows:

Deflection Measuring Micrometer:

This has been described in detail in a previous publication (**3**). Briefly, the upper one of a pair of contacts was fastened to the specimen end of a bearing housing on the R. R. Moore machine, the lower contact being carried on a micrometer screw mounted on the bed of the machine. The contacts operated a signalling device through a tube circuit, so the position of the upper contact could be determined by raising the lower contact and noting the reading of the micrometer drum when the circuit closed. The contacts were set slightly apart so that any increase in the deflection of the specimen during test would close the circuit, operating the signal. The best results with this apparatus were obtained by mounting the fatigue testing machine on springs to minimize vibrations from extraneous sources. No specimen was used which caused excessive vibration when the machine was running. With these precautions, it was possible to detect deflections of 0.001 to 0.002 mm.

Stroboscopic Viewing Device:

One of a pair of contacts was mounted in a bakelite disk and fitted to the shaft of the R. R. Moore machine, the other (stationary) contact being mounted so as to be rotatable about the center line of the machine. These contacts were used to flash a stroboscope and by adjusting the stationary contact circumerentially the flash could be made to occur at any position in the revolution of the specimen. The flashing light was directed, by means of a condenser and mirror, onto the bottom surface of the specimen, and a low-power microscope fitted with a prism in front of the objective was used to view the illuminated area. Because this area was stressed in tension, the fatigue cracks were opened and made readily visible.

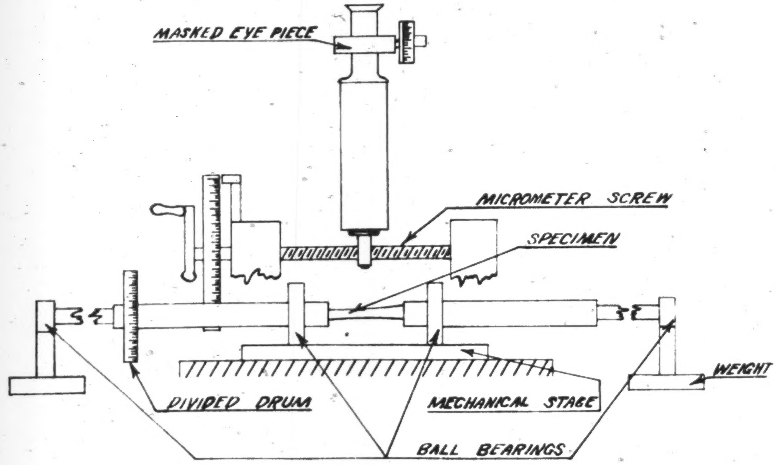

FIG. 2.—Equipment for Measuring and Counting Fatigue Cracks.

Apparatus for Counting and Measuring Fatigue Cracks:

A traveling microscope was used for measuring fatigue cracks, as sketched in Fig. 2. The specimen was removed from the fatigue machine and mounted in spindles on the stage so that it was parallel to the traversing screw of the microscope. Weights hung on the ends of the spindles produced a bending moment which stressed the upper surface in tension. The area being examined was illuminated by an intense beam incident at a grazing angle in the plane of the axes of the specimen and microscope. Since the polishing scratches seen under the microscope were also in this plane, little light was reflected into the microscope. A fatigue crack, being at right angles to the incident beam, showed up brightly against the generally dark background.

The eyepiece of the microscope was fitted with both a cross-hair and an adjustable mask. For measuring the length of cracks a subdivided drum was fitted on one of the spindles. The central angle subtended by the crack was obtained by rotating the specimen under the cross-hair and noting the drum reading at each end of the crack. For counting cracks the mask in the eyepiece was set so that the field observed on the specimen was just 1 mm. wide (the edges of the mask were at right angles to the length of the specimen). The surface could then be scanned in 1-mm. wide bands, starting from the reference mark at the minimum secton, by traversing the microscope 1 mm. after each revolution of the specimen. A ratchet was set to give an audible click to indicate the completion of each revolution.

TEST RESULTS WITH NOTCHED SPECIMENS

Previous work, both at this Bureau and elsewhere, revealed two significant factors for satisfactorily evaluating fatigue damage. First, the dispersion of the experimental results necessitates an average of about eight specimens to give a sufficiently precise result for any one condition. Second, the evaluation of damage by running tests to complete fracture is limited to cycle ratios[3] sufficiently small so that the chance of crack formation during pre-stressing is negligible; otherwise the damage in some cases would be dependent only on the size of an initial fatigue crack rather than on the effect of pre-stressing.

The methods followed in the study of fatigue damage with notched specimens were chosen on the basis of these considerations. In order to guard against crack formation it was necessary to have a means of detecting fatigue cracks at an early stage without stopping the test. Such a method was developed based on the fact that the deflection of a rotating-beam specimen under constant load increases when a crack forms. The apparatus and technique used to detect and measure this deflection are described in the preceding section of this report. The deflection is a function of the size of crack, and the criterion of failure chosen was a deflection of 0.005 mm., corresponding to a crack area about 12 per cent of the original cross-section (**3**). The specimens were actually run until the deflection had increased to 0.01 mm. in order to make certain that the deflection was due to a crack, but the data in this section are based on the number of cycles run at the time the specimen deflected 0.005 mm. This number is referred to as N_c.

Tests were conducted in the conventional manner (to fracture) on a group of notched specimens to determine the *S-N* curve and the fatigue limit. Four stresses in the range between the fatigue limit (39 000 psi. for notched specimens) and the yield strength of the steel (62 500 psi.) were used in the investigation. The four stresses chosen were 42 000, 48 000, 54 000, and 60 000 psi.

Tests were made to determine N_c at each of these four stresses. At least ten specimens were tested at each stress and the median of each group was used as the value of endurance at that stress. The median was considered to be more representative of a group of endurance values than the average, as it is less affected by an occasional excessively divergent value. Also the median can be determined in cases where some elements of the group cannot be expressed numerically but are known to lie in a certain range. For example, in determining the endurance at a given stress, if only one specimen of the group did not fail, the average endurance of the group would be infinite, whereas the median value would be finite.

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[3] Cycle ratio is defined as the ratio of the number of cycles run at a given overstress to the number necessary to cause failure at that stress.

Since the scatter of the results was not uniform in the four groups, it was necessary to test more specimens in some cases than in others in order to obtain approximately the same precision for each value of N_c. The number of tests in each group ranged from 11 to 17.

TABLE II.—ENDURANCE OF NOTCHED SPECIMENS.

Stress, psi.	Number of Cycles to Crack
±42 000	963 000
±48 000	264 000
±54 000	93 000
±60 000	44 000

TABLE III.—DAMAGE TEST CONDITIONS.

Pre-Stress, psi.	Test Stress, psi.
±42 000 ±54 000	±48 000
±48 000 ±60 000	±54 000

The values of N_c (the number of cycles to form a crack of definite size) for each selected stress are listed in Table II.

In the next stage of the investigation each specimen was first stressed for a predetermined number of cycles at one of the four chosen stresses, then the stress was changed as given in Table III and the number of cycles to failure determined. The difference between this value and the median value of endurance for the original material at the same stress (Table II) gave a measure of the damage caused by the pre-stressing. This difference was expressed as a percentage of the undamaged endurance.

For each of the combinations in Table III, tests were made with the pre-stressing carried to 10, 25, 50, 75, and 90 per cent of the median value of N_c. The number of tests made for each pre-stress condition was between six and ten depending on the scatter of the individual values. The median values for each group are listed in Table IV.[4] The variation of damage with percentage pre-stress is shown graphically in Figs. 3 and 4 for a test stress of ±48,000 psi., and in Figs. 5 and 6 for a test stress of ±54,000 psi. In the graphs the broken

TABLE IV.—EFFECT OF PRE-STRESS ON ENDURANCE OF NOTCHED SPECIMENS OF S.A.E. X4130 STEEL STRESSED AS ROTATING BEAMS.

Number of Specimens	Test Stress, psi.	Pre-Stress, psi.	Cycle Ratio, per cent of N_c value	Endurance, cycles	Damage, per cent	SIQ, per cent[a]
17	±42 000		0	963 000	0	25
11	±48 000		0	264 000	0	13
7		±42 000	10	198 000	25	12.5
10		±42 000	25	220 000	16.7	18
10		±42 000	50	192 000	27.3	20
10		±42 000	75	177 000	32.9	26
10		±42 000	90	11 000	95.8	...
8	±48 000	±54 000	10	205 000	22.3	14
7		±54 000	25	125 000	52.6	12
7		±54 000	50	72 000	72.7	9
7		±54 000	75	50 000	81.1	15
8		±54 000	90	12 000	95.5	...
17	±54 000	....	0	93 000	0	26
8		±48 000	10	87 000	6.4	23
6		±48 000	25	73 000	21.5	10
8		±48 000	50	59 000	36.6	16
8		±48 000	75	27 000	71	...
8		±48 000	90	3 000	96.8	...
15	±54 000	±60 000	10	84 000	9.7	21
7		±60 000	25	63 000	32.2	14
6		±60 000	50	31 000	66.7	10
8		±60 000	75	15 000	83.9	12
8		±60 000	90	3 000	96.8	...
15	±60 000	....	0	44 000	0	23

[a] SIQ = Semi-interquartile range.

---

[4] The middle number of a series arranged in order of magnitude is the median. The middle one of the numbers that lie below the median is the lower quartile, the middle one of those above is the upper quartile. Half the difference between the upper and lower quartiles is the semi-interquartile (SIQ) range. This is a measure of the dispersion of the individual values which expresses the same limits relative to the median as the probable error expresses relative to the average.

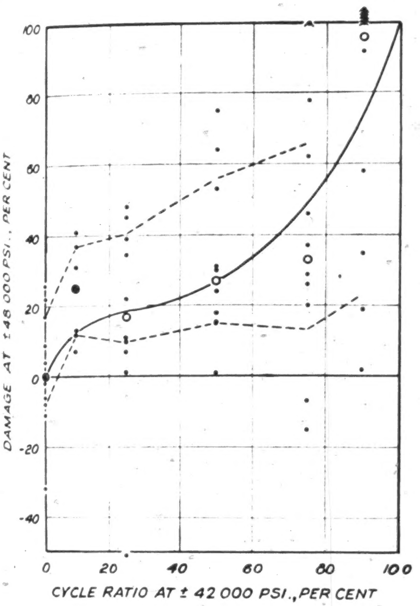

FIG. 3.—Damage at ±48,000 psi. Due to Stressing at ±42,000 psi. Notched specimens. Cycle ratios and damage based on N_c.

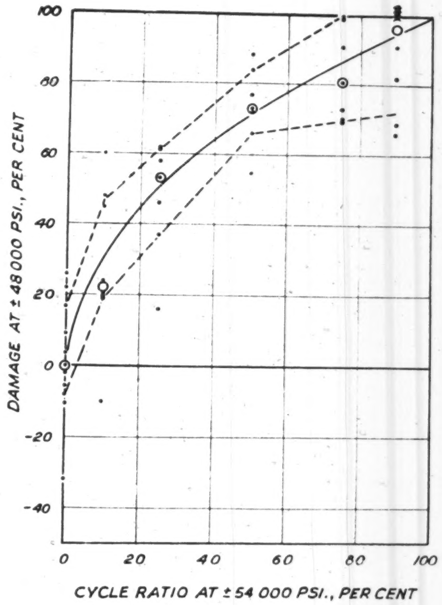

FIG. 4.—Damage at ±48,000 psi. Due to Stressing at ±54,000 psi. Notched specimens. Cycle ratios and damage based on N_c.

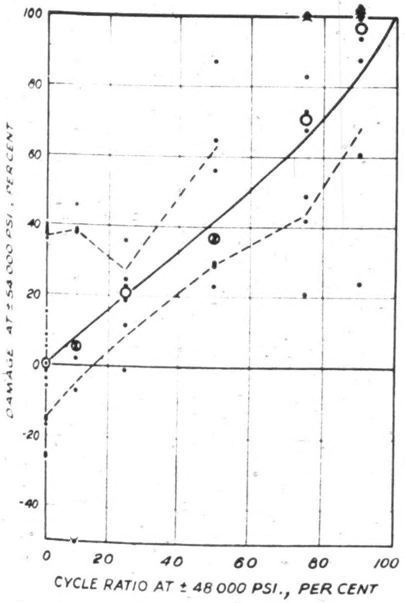

FIG. 5.—Damage at ±54,000 psi. Due to Stressing at ±48,000 psi. Notched specimens. Cycle ratios and damage based on N_c.

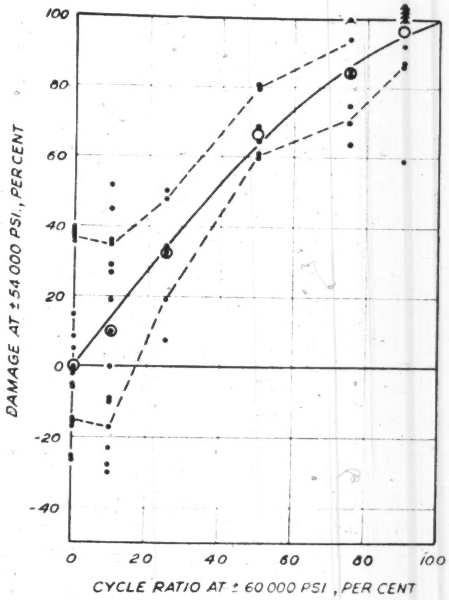

FIG. 6.—Damage at ±54,000 psi. Due to Stressing at ±60,000 psi. Notched specimens. Cycle ratios and damage based on N_c.

lines join the upper and lower quartile[4] points of each group of values and thus give an indication of the scatter of the data. The curves from Figs. 3 to 6 are combined in Fig. 7 for comparison.

In the test groups in which pre-stressing was carried to 75 and 90 per cent of the endurance, some specimens failed before the pre-stressing was completed, that is, the damage was greater than 100 per cent. The inclusion of these results caused no difficulty in determining the median, although it did make the comparison of precision between the groups more uncertain.

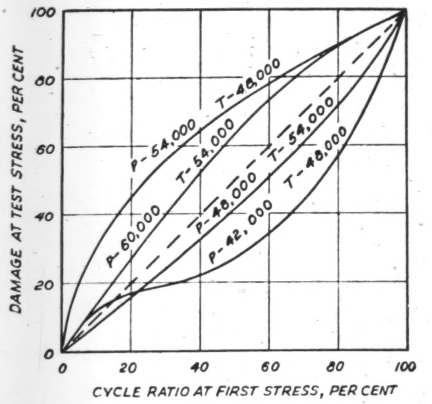

FIG. 7.—Damage as Measured by Loss of Endurance (N_e). Curves from Figs. 3 to 6, notched specimens.

P = Pre-stress in pounds per square inch.
T = Test stress in pounds per square inch.

While the results of the damage tests as shown in Fig. 7 are not directly comparable with those obtained with smooth specimens (**1**, **2**), there are certain similarities. The tendency with both types of specimen was for the curve representing a pre-stress above the test stress to lie above the 45-deg. line. The opposite tendency was noted when the magnitude of the pre-stress was below that of the test stress. (The 45-deg. line may be considered as the damage curve for a pre-stress equal to the test stress.)

In order to provide a basis of comparison with the results of fatigue damage reported by others (usually based on the fatigue limit), some tests of this type were conducted with two values of pre-stress, ±42,000 and ±60,000 psi. The effect of various amounts of pre-stressing on the fatigue limit subsequently determined by fracture tests is shown in Table V and Fig. 8. The number of cycles of pre-stress used for the 75 and 90 per cent cycle ratios was the same as the number used in the previous phase of the investigation, but a correction was made in determining the cycle ratios represented by these amounts of pre-stressing. The method of making this correction will be shown for the case of the highest cycle ratio at ± 42,000 psi.

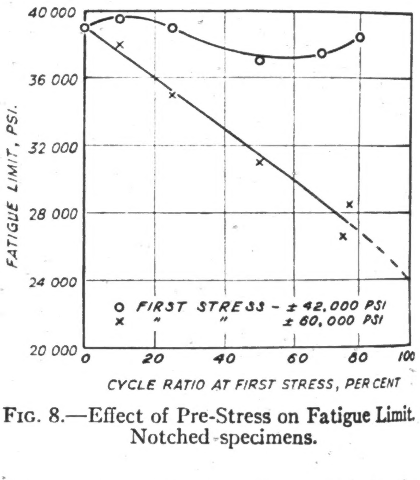

FIG. 8.—Effect of Pre-Stress on Fatigue Limit. Notched specimens.

The medium N_c for this stress was 963×10^3. When it was desired to pre-stress a group of specimens to 90 per cent of this value, or 867×10^3 cycles, some of the specimens failed before pre-stressing was completed. Only those which did not fail in pre-stressing could be used for the determination of fatigue limit, so this determination was not truly representative. Therefore, in the original series of values of N_c, the median of all values greater than 867 × 10^3 was taken as being representative of the specimens which were used to determine the fatigue limit after pre-stressing. The cycle ratio as plotted in Fig. 8 is based on this second median (1057 × 10^3 cycles) and is consequently less than 90 per cent. A similar correction was made for the 75 per cent prestress at ±42,000 psi., and for the 90 per cent value at ±60,000 psi. The corrected values of cycle ratio at ±42,000 psi. were 68 and 80 per cent; at ±60,000 psi. the highest value was 77 per cent. The slight repair of damage above 50 per cent cycle ratio during stressing at ±42,000 psi., if real, is surprising, and should be verified by additional work.

TABLE V.—EFFECT OF PRE-STRESS ON FATIGUE LIMIT OF NOTCHED SPECIMENS OF S.A.E. X4130 STEEL STRESSED AS ROTATING BEAMS.

Pre-stress, ±42,000 psi.			Pre-stress, ±60,000 psi.		
Cycle Ratio, per cent	Fatigue Limit, psi.	Damage, per cent of maximum	Cycle Ratio, per cent	Fatigue Limit, psi.	Damage, per cent of maximum
10	±39 500	−3	10	±38 000	6
25	±39 000	0	25	±35 000	26
50	±37 000	13	50	±31 000	52
68	±37 500	10	75	±26 500	81
80	±38 500	3	77	±28 500	68

In order to make the data on the effect of pre-stressing on fatigue limit (Fig. 8) more directly comparable with those on the effect of pre-stressing on endurance (N_c, Fig. 7) an attempt was made to determine the fatigue limit of cracked specimens. The accurate calculation of stress on a specimen containing a fatigue crack is virtually impossible because of the irregular and unpredictable shape of the crack. A rough estimate of the increase in stress with spreading of the crack was made by assuming the stress inversely proportional to the ratio of uncracked area to original area.

The specimens used for this determination had deflected 0.010 mm. in the fatigue test. This corresponded to a crack area equal to 17 per cent of the original minimum section and the fatigue limit of the cracked specimen was found to be ±23,500 psi. In Fig. 9 the data

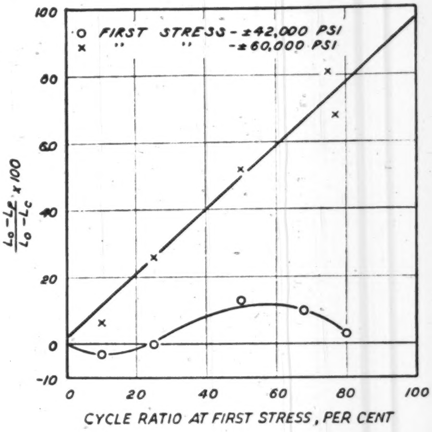

FIG. 9.—Damage as Measured by Loss of Fatigue Limit, Notched Specimens.

L_o = Fatigue limit of original specimen.
L_p = Fatigue limit of pre-stressed specimen.
L_c = Fatigue limit of cracked specimen.

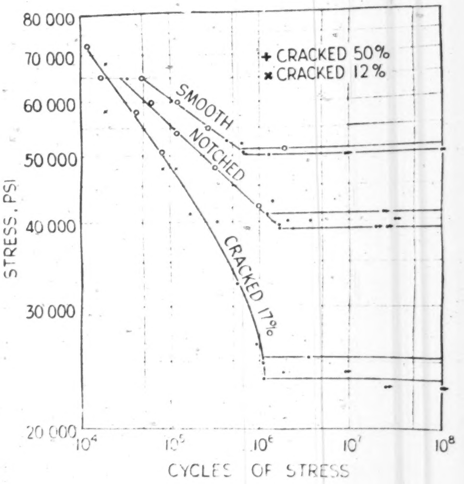

FIG. 10.—*S–N* Curves for S.A.E. X4130 Steel Under Three Conditions of Stress Concentration. Large open points represent the median values from a group of specimens tested at the same stress. Small closed points are the results of individual tests.

of Fig. 8 have been replotted with the damage (decrease of fatigue limit) expressed as percentage of the damage caused by a crack of this size. The straight line drawn through the data points for ±60,000 psi. was determined by the method of least squares.

Figure 10 shows the *S-N* curves for specimens with three types of stress concentration: smooth (zero concentration), notched, and cracked (maximum concentration). The points marked X or + in Fig. 10 are the results from cracked specimens having crack areas other than 17 per cent. It will be noted that the value for a specimen having an area ratio of 50 per cent lies very far above the 17 per cent ratio curves while the values for specimens having a 12 per cent ratio are in the same range or lower. This suggests that the stress concentration at high stresses is less for large cracks than for small cracks. Much more experimental work would be required before any general statement could be made with certainty.

TABLE VI.—STRESS-CONCENTRATION FACTORS IN SPECIMENS OF S.A.E. X4130 STEEL STRESSED AS ROTATING BEAMS.

Type of Specimen	Fatigue Limit, psi.	S.C.F.[a] from Fatigue Limit	Slope of *S-N* Curve	S.C.F.[a] from Slope	Theoretical S.C.F.[a]
Smooth	±50 000	1.0	−0.767	1.0	1.0
Notched	±39 000	1.28	−0.983	1.28	1.59
Cracked, 17 per cent	±23 500	2.13	−1.46	1.91	...

[a] S.C.F. = Stress-concentration factor.

In Table VI are shown the values of stress-concentration factors given by the ratios of the fatigue limits of the specimens under three conditions of stress concentration. Almen (**4**) has shown that the slope of the falling part of the *S-N* curve increases with increasing stress concentration. If the slope of the falling part of the *S-N* curve is a measure of effective stress concentration, then the ratios of the slopes of the three lines of Fig. 10 should give the same values for these factors as obtained from the fatigue limits. There was substantial agreement of these factors as determined by the two methods (Table VI). The value of theoretical stress-concentration factor given in Table VI for the notched specimen was calculated from a table given by Roark (**5**).

TEST RESULTS WITH SMOOTH SPECIMENS

Methods of Estimating the Start of Cracking:

The preceding experiments showed that fatigue-damage results are more reproducible when based on the number of cycles to form a small crack than when based on the number of cycles to fracture. It was considered that even greater reproducibility might be obtained if it were possible to determine the number of cycles at which a crack first starts. Then the damaging cycle ratios could be based on the pre-crack stage only. This was done in the experiments described below, all tests being made on smooth ($9\frac{7}{8}$-in. contour radius) specimens.

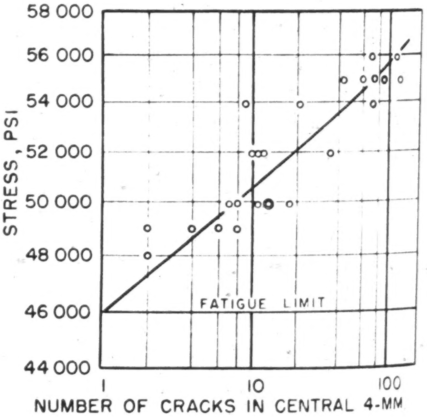

FIG. 11.—Influence of Applied Stress on the Number of Cracks Formed Prior to Fracture.

In the discussion of methods for estimating the start of cracking, the relationship shown in Fig. 11 should be kept in mind. This shows a semilogarithmic plot of test stress against the number of cracks formed prior to fracture. The points plotted are based only on cracks lying in a band 4 mm. wide at the center of the specimen. The number of cracks increased rapidly as the stress was raised above the fatigue limit. Because of the large variation in the number of cracks formed, two different methods were used for estimating the start of cracking, depending on the number of cracks observed.

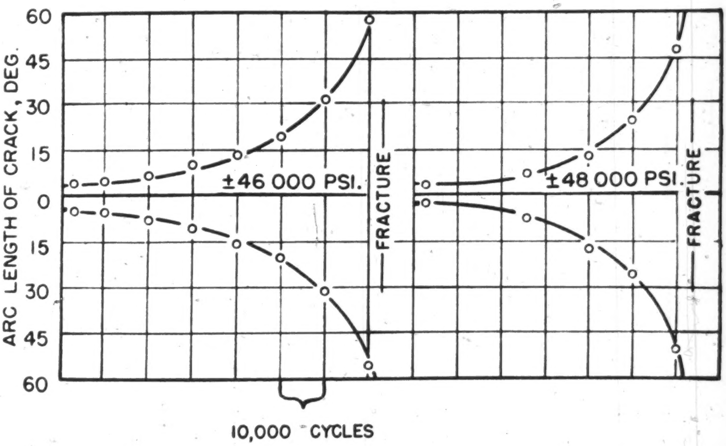

FIG. 12.—Curves Showing Change of Crack Length at Each of Two Different Stresses. The mid point of the crack when first measured is taken as zero.

By means of the stroboscopic apparatus described in the section on Testing Machines and Special Apparatus, the surface of a specimen was observed during the tests. The field of the microscope was large enough so that the chance of a crack forming outside the field was small. By adjusting the stationary contact any point on the circumference could be brought into view, thus permitting observation of the initial crack irrespective of circumferential position. A small crack could be seen readily on the surface when in bottom position because it was opened up by the tensile stress.

As soon as a crack was observed, the test was stopped and the circumferential length of the crack was measured. This procedure was repeated at frequent intervals until fracture occurred. Figure 12 shows the growth of two cracks each under a different stress. It will be noted that the curves appear to start from a finite value of crack length, rather than from zero. This is a surprising observation, and one which should be checked further, but in the present investigation there appeared to be no question of its validity, so all extrapolation was performed on this basis. The fact that small, though numerous cracks frequently have been observed at stresses considerably in excess of the fatigue limit suggests that the large value of the initial crack length shown in Fig. 12 is associated with stresses nearer the fatigue limit.

It was found that the crack length L could be expressed by the equation

$$\log (L - C) = \alpha N \quad (1)$$

where C and α are constants, the latter dependent on stress, and N the number of cycles. This is shown graphically in Fig. 13 where $L - C$ is plotted logarithmically against *N* for one of the curves shown in Fig. 12. When curves similar to Fig. 13 were plotted for various nominal stress values, it was found that the slope was a simple function of the stress, as shown in Fig. 14.

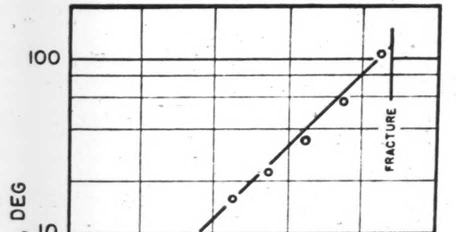

FIG. 13.—Semi-Logarithmic Plot of Crack-Growth Curve. The ordinate is the arc length (L) of the crack minus a constant (C).

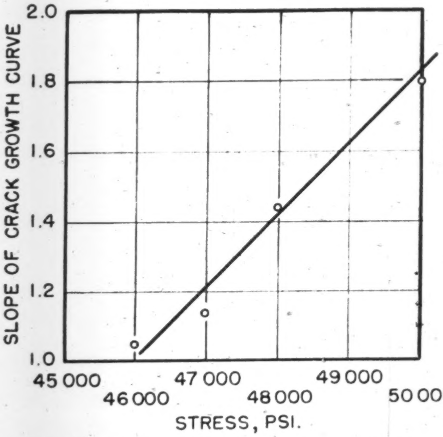

FIG. 14.—Influence of Stress on the Rate of Crack Growth.

Curves of the type shown in Fig. 13 were used to determine by extrapolation the point at which cracking starts, assuming that the cracks grow exponentially from an original arc length of about 6 deg. as indicated in Fig. 12. The deflection measuring apparatus described earlier in this report was used to indicate the presence of a crack, but this did not operate until the crack reached 50 or 60 deg. in circumferential length, the growth curves (Fig. 13) being used to extrapolate back to the start of the crack.

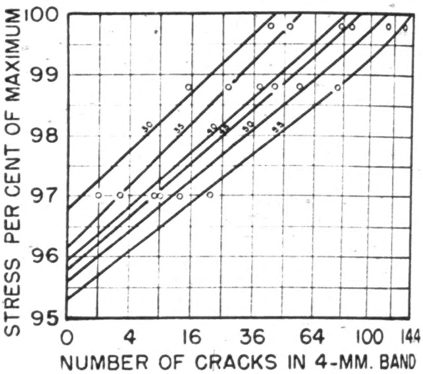

FIG. 15.—Results of Crack Counts on One Smooth Specimen at Various Stages Between the Beginning of Cracking and Fracture. Numbers on curves represent thousands of cycles of stress which the specimen had run when the cracks were counted. Total fracture occurred at 59,000 cycles.

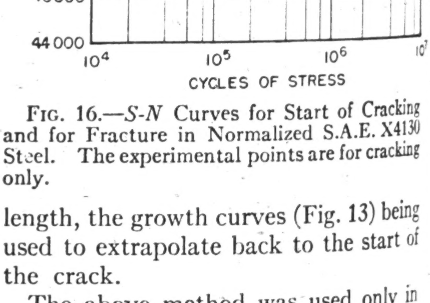

FIG. 16.—*S-N* Curves for Start of Cracking and for Fracture in Normalized S.A.E. X4130 Steel. The experimental points are for cracking only.

The above method was used only in cases where the total number of cracks was not more than five, since more numerous cracks might change the surface stress sufficiently to invalidate the extrapolation. For specimens which had numerous cracks the extrapolation was performed as follows: by means of the masked eyepiece described previously the cracks were counted in circumferential bands 1 mm. wide. Practically all of the cracks occurred within 6 mm. of the center of the specimen and for the purpose of totaling the number of cracks this area was divided into three zones. The first extended 2 mm. on each side of the center, the second from 2 to 4 mm. on each side and the third from 4 to 6 mm. The average stress in each zone was calculated from the geometry of the specimen and found to be 99.8, 98.8, and 97.0 per cent of the stress at the center for the first, second, and third zones, respectively. The square root of the number of cracks in a zone plotted against the average stress in that zone, gave a nearly linear relationship which could be extrapolated back to the stress at which no cracks would be formed. Curves of this type are shown in Fig. 15.

TABLE VII.—PRE-STRESS CONDITIONS.

Stress, psi.	Cycle Ratio, per cent of N_e
±48 000	10
	33⅓
	66⅔
	90
±54 000	10
	33⅓
	66⅔
	90

TABLE VIII. RESULTS OF DAMAGE TESTS.

Cycle ratios are based on N_e. "F" indicates that specimen fractured; "U" indicates that the specimen did not break.

Pre-stress, ±48 000 psi.		Pre-stress, ±48 000 psi.	
Stress, psi.	10^3 Cycles	Stress, psi.	10^3 Cycles
CYCLE RATIO, 10 PER CENT		CYCLE RATIO, 66⅔ PER CENT	
±54 000	102 F	±54 000	73 F
±54 000	70 F	±54 000	63 F
±54 000	96 F	±54 000	131 F
±52 000	225 F	±52 000	129 F
±50 000	146 F	±50 000	96 F
±46 200	1 187 F	±46 000	1 131 F
±46 200	514 F	±46 000	516 F
±46 000	22 339 U	±44 600	240 F
±46 000	27 721 U	±44 400	1 038 F
±45 800	44 988 U	±44 400	20 635 U
		±44 200	36 233 U
CYCLE RATIO, 33⅓ PER CENT		CYCLE RATIO, 90 PER CENT	
±54 000	61 F	±54 000	99 F
±54 000	81 F	±54 000	24 F
±54 000	142 F	±54 000	58 F
±52 000	86 F	±52 000	112 F
±50 000	141 F	±50 000	111 F
±47 000	313 F	±46 000	622 F
±46 000	743 F	±44 000	132 F
±45 300	1 608 F	±42 600	410 F
±45 100	22 711 U	±42 400	416 F
±45 000	831 F	±42 200	34 516 U
±44 900	40 787 U	±42 000	17 709 U
±44 800	21 942 U		
±44 500	20 027 U		
Pre-stress, ±54 000 psi.		Pre-stress, ±54 000 psi.	
Stress, psi.	10^3 Cycles	Stress, psi.	10^3 Cycles
CYCLE RATIO, 10 PER CENT		CYCLE RATIO, 66⅔ PER CENT	
±52 000	110 F	±52 000	69 F
±50 000	318 F	±50 000	186 F
±48 000	720 F	±48 000	132 F
±45 800	2 003 F	±48 000	86 F
±45 800	834 F	±46 000	454 F
±45 600	33 016 U	±44 000	19 227 U
±45 600	428 F	±44 000	19 133 U
±45 400	21 033 U	±43 000	18 986 U
		±42 000	459 F
		±40 400	16 101 U
CYCLE RATIO, 33⅓ PER CENT		CYCLE RATIO, 90 PER CENT	
±52 000	79 F	±59 000	12 F
±50 000	109 F	±52 000	54 F
±48 000	267 F	±50 000	91 F
±46 000	1 276 F	±48 000	128 F
±45 600	327 F	±45 000	641 F
±44 000	853 F	±44 000	162 F
±43 800	23 882 U	±43 000	25 304 U
±43 800	19 229 U	±42 000	144 F
±43 600	21 451 U	±42 000	16 009 U
		±41 000	575 F
		±40 400	176 F
		±40 200	249 F
		±40 000	21 338 U
		±40 000	11 643 U

Points obtained for the start of cracking by these two methods of extrapolation (Figs. 13 and 15) are shown on Fig. 16, both the stress and number of cycles being plotted logarithmically. The straight line through the points was determined by the method of least squares. A similar graph based on the points representing fracture is also included in this figure (the points for this curve are not shown).

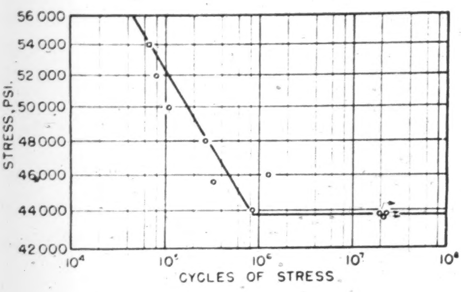

Fig. 17.—*S-N* Curve for Specimens Which Had Been Subjected to $33\frac{1}{3}$ per cent Cycle Ratio at ±54,000 psi. The square point is at the stress used for pre-stressing and the number of cycles obtained by subtracting the pre-stress cycles from expected life at this stress.

Damage Tests Including Strength-Loss Line Hypothesis:

In the next phase of the work, two stresses were chosen between the fatigue limit and the yield strength of the material, and specimens were stressed for various cycle ratios at these stresses. The stresses chosen were ±48,000 and ±54,000 psi., and the cycle ratios were based on the number of cycles to the start of cracking.[5] Eight pre-stress conditions were used, as shown in Table VII.

The pre-stressed specimens were used to determine a complete *S-N* curve for each condition, a typical curve of this type being shown in Fig. 17. This method has been used recently by Kommers (2) to measure fatigue damage. These curves were determined in the usual manner, that is by tests to fracture. The experimental data are shown in Table VIII. The curves for the eight pre-stress conditions are shown in Figs. 18 and 19. The cycle ratio for the largest number of cycles should be less than 90 per cent because of the specimens which cracked before reaching the pre-determined number of cycles. However, unlike the tests described in the preceding section, these results could not be accurately corrected for the premature failures, so no attempt was made to do so.

An additional curve is shown on Fig. 18, representing the *S-N* relationship for specimens which had been improved by understressing. Only five specimens were used to determine this curve, and the pre-stress treatments in the five cases were not identical, but as the scatter appeared to be small, the curve is probably reasonably reliable. The complete history of each of the five specimens is given in Table IX.

TABLE IX.—TESTS OF SPECIMENS IMPROVED BY UNDERSTRESSING.

"F" indicates that specimen fractured, "U" indicates that specimen did not fracture.

Pre-stress, psi.	10^3 Cycles at Pre-stress	Test Stress, psi.	10^3 Cycles at Test Stress
±45 000	21 700	±56 000	80 F
±46 000	24 239	±56 000	79 F
±45 900	18 567	±54 000	144 F
±45 900	26 700	±52 000	438 F
±45 800	31 578	±50 000	3933 U

In Fig. 19, a dashed line represents the *S-N* relationship for specimens which had been damaged 100 per cent; that is, specimens which were just ready to crack. This curve was obtained as follows: points were obtained at ±48,000 and ±54,000 psi. by subtracting the number of cycles to form a crack from the total number to fracture. Two additional points were obtained at ±42,000 and ±38,000 psi. by testing specimens having very small cracks (2 to 3 deg. long). The four points obtained in this way fell very close to the straight line shown in Fig. 19. There is no way to obtain directly the fatigue limit for 100 per cent damaged specimens.

It will be noted in Figs. 18 and 19 that the curves appear to radiate from approximately the same point. This

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[5] The term N_e will be used to designate the number of cycles to the start of cracking. This value is determined from the cracking curve of Fig. 16.

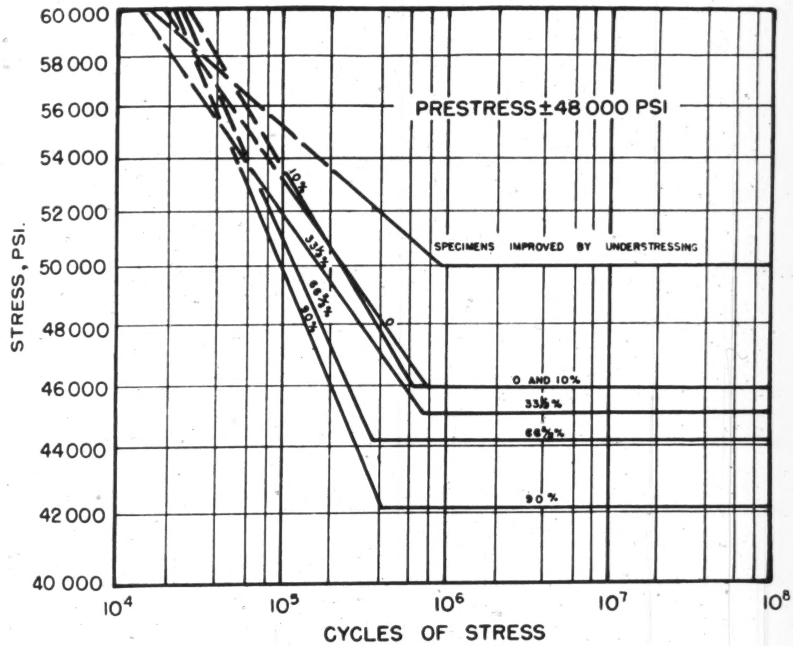

FIG. 18.—*S-N* Curves for Specimens Overstressed at ±48,000 psi. and for Specimens Improved by Understressing. The figures on the curves represent the cycle ratio (based on N_e) to which the specimens were run in pre-stress.

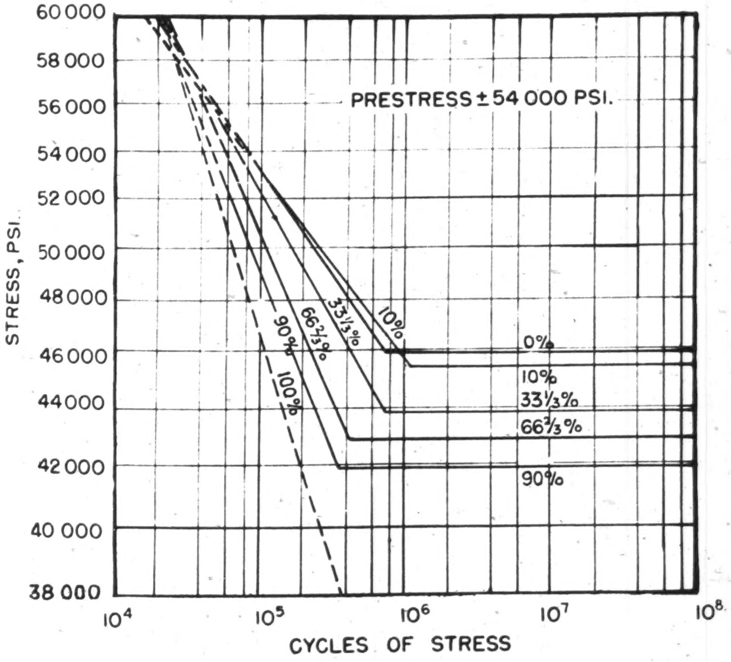

FIG. 19.—*S-N* Curves for Specimens Overstressed at ±54,000 psi. The figures on the curves represent the cycle ratios (based on N_e) to which the specimens were run in pre-stress.

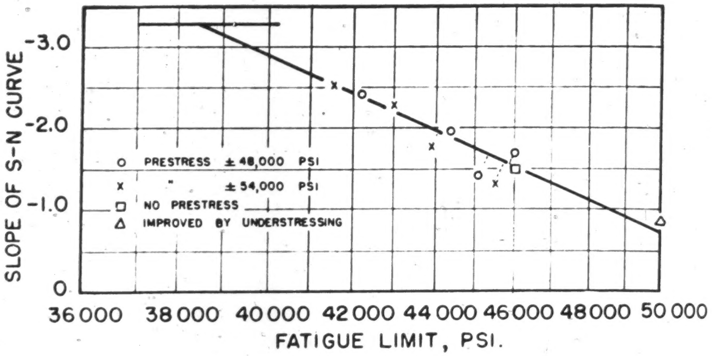

FIG. 20.—Strength-Loss Relationship Derived from Curves of Figs. 18 and 19.

suggests that the damage could be measured by a simple geometrical property of the *S-N* curves. In Fig. 20 the logarithm of the fatigue limit is plotted against the slope of the falling part of the *S-N* curve. While there is considerable scatter, the points lie approximately on a straight line, indicating that the damage is of the same nature regardless of the stress at which it was inflicted. The horizontal line at 3.27 represents the slope of the 100 per cent damage curve and the intersection of this line with that through the points should serve as a means of estimating the fatigue limit of the 100 per cent damaged specimens. There appears to be no method for checking the value obtained.

It will be noted that the point representing specimens which had been improved by understressing lies near the straight line through the damage points. This indicates that the changes due to understressing are of a similar type to those occurring on overstressing, but the results produced are in the opposite direction.

If the type of damage does not depend on the stress at which it is inflicted, then the amount of damage should be represented by distance along the curve of Fig. 20, being expressed as per cent of the distance from the value for the original material to that for 100 per cent damage. This line therefore will be referred to as the strength-loss line. The distance along this line is plotted in Fig. 21 against the cycle ratio for the two values of pre-stress used, the distance being measured to the foot of the perpendicular from each point to the line. The curves of Fig. 21 are significant, since they permit the prediction of the complete *S-N* curves for specimens which have been damaged by any amount of pre-stress at ±48,000, ±54,000 psi., or by any combination of these stresses.

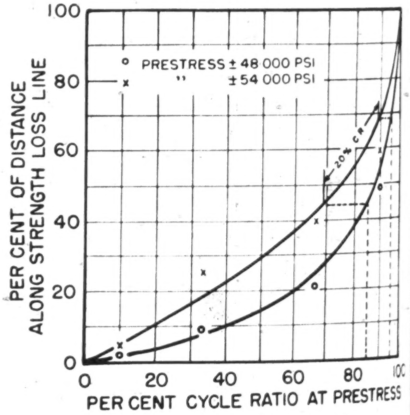

FIG. 21.—Cycle Ratio *versus* Damage Relationship for Two Stresses. Dashed lines show method of predicting results for one complex loading schedule.

Multiple Pre-Stress Tests:

In order to investigate the reliability of the predictions based on the curves of Fig. 21 and at the same time to study the damage caused by more complex loading programs, six pre-stressing schedules shown in Table X were used. For each schedule, three to five specimens were tested to fracture at the test stress, and for schedule IV an attempt was also made to determine the fatigue limit after pre-stress. All results of these tests are shown in Table X, together with the predictions based on Fig. 21. The method of making these predictions is illustrated by the dotted lines on the figure, showing the steps involved in schedule V. 85 per cent cycle ratio at ±48,000 psi. results in 44 per cent damage; 44 per cent damage corresponds to 69 per cent cycle ratio at ±54,000 psi; 69 + 20 = 89 per cent cycle ratio at ±54,000 psi., which corresponds to 69 per cent damage; 69 per cent damage would be caused by 96 per cent cycle ratio at ±48,000 psi., so 4 per cent cycle ratio should remain after the pre-stressing is complete. The number of cycles to the start of cracking at this stress is 333,000. 0.04 × 333,000 = 13,000, which represents the number of cycles to form a crack at the test stress. In addition, it requires 78,000 cycles at this stress for the crack to grow to fracture, so the total predicted life under these conditions is 91,000 cycles.

TABLE X.—MULTIPLE PRE-STRESS TESTS.

Pre-stress Schedule[a]	Test Results, 10^3 Cycles to Fracture					Median 10^3 Cycles	Prediction 10^3 Cycles
I	126	304	163	147	207	163	177
II	65	81	57	37	21	57	56
III	7	28	5			7	34
IV[b]	174	80	101			101	148
V	<0	98	<0	102	86	86	91
VI	106	266	75			106	137

[a] I.—33⅓ per cent cycle ratio at ±48 000 psi. + 33⅓ per cent cycle ratio at ±54 000 psi., test ±48 000 psi.

II.—33⅓ per cent cycle ratio at ±54 000 psi. + 33⅓ per cent cycle ratio at ±48 000 psi., test ±54 000 psi.

III.—75 per cent cycle ratio at ±54 000 psi. + 15 per cent cycle ratio at ±48 000 psi., test ±54 000 psi.

IV.—50 per cent cycle ratio at ±48 000 psi. + 33⅓ per cent cycle ratio at ±54 000 psi., test ±48 000 psi.

V.—85 per cent cycle ratio at ±48 000 psi. + 20 per cent cycle ratio at ±54 000 psi., test ±48 000 psi.

VI.—15 per cent cycle ratio at ±48 000 psi., + 10 per cent cycle ratio at ±54 000 psi., then repeat twice more. Test ±48 000 psi.

[b] Determination of fatigue limit for schedule IV.

"F" indicates that specimen fractured, "U" indicates that specimen did not break.

In addition to those listed, three specimens cracked before the pre-stressing was completed.

Stress, psi.	10^3 Cycles	Fatigue-Limit Prediction
±42 000	4 898 U	±43 200 psi.
±42 200	22 715 U	
±42 800	5 459 U	
±43 000	399 F	

The results listed in Table X show reasonable agreement with the predictions, considering the large probable error of the tests involved. A small change in the rate of damage from one specimen to the next, particularly in the region where the curves of Fig. 21 are steep, may cause a large variation in the damage due to a certain pre-stress. Other methods tried for predicting these results, including the simple cumulative addition of cycle ratios suggested by Miner (6), failed to give reasonably close values for more than one or two of the schedules.

Repair of Damage:

Two exploratory tests were made to verify the work of others on the repair of fatigue damage. In each case the pre-stress was 90 per cent cycle ratio (based on N_s) at ±48,000 psi. The first specimen was then stressed at ±42,200 psi. (just under the new fatigue limit) for 34 × 10^6 cycles. On testing at ±48,000 psi. this specimen did not fail in 40 × 10^6 cycles. The understressing had not only repaired the prior damage but raised the fatigue limit above that of the material in its original condition. As a matter of interest this same specimen was subsequently "coaxed" by stressing 20 to 30 million cycles at each of seven successively higher stresses. At a stress of ±62,000 psi. the specimen endured 21 × 10^6 cycles, then failed after 2,500,000 cycles at ±64,000 psi. Thus, a specimen, damaged to the extent where the fatigue limit was less than ±42,500 psi. was improved by suitable stress treatment, bringing its fatigue limit above ±62,000 psi.

The second damaged specimen was normalized in vacuum, reproducing as closely as possible the temperature cycle of the original heat treatment. On testing at ±48,000 psi. the specimen fractured in 126,000 cycles. The normal expectancy for specimens after this prestress would be 143,000 cycles, indicating that the heat treatment resulted in no repair of the fatigue damage.

DISCUSSION

The life of a specimen under fatigue stressing consists of two distinct phases: during the first, part of the material is progressively damaged until a small crack is formed, and during the second, this crack grows until fracture occurs. In this report the term fatigue damage is used to designate only the change occurring during the first stage, material being considered 100 per cent damaged at the time the first crack forms. If one wishes to study the first-stage damage, some method of determining the start of cracking is required. Lacking this, the effects of the second stage will confuse the results. Within the stress range used in this study, the proportion of the total life represented by the second stage varies from as much as 50 to as little as 10 per cent, so it can be seen that a large error would be involved if the two stages were not separated.

With the S.A.E. X4130 steel used in this investigation, the ratio of the second stage to the total life of the specimen was larger at stresses considerably above the fatigue limit than at stresses close to the fatigue limit. This is contrary to what certain other investigators have reported, but such a discrepancy may be expected, since the ratio depends on many factors such as the slope of the *S-N* curve for the original material, the rate of crack propagation in the metal, and the size of the specimen.

The damage resulting from a particular number of cycles of overstress depends on the past history of the specimen. Thus, the performance cannot be predicted accurately for metals in service which are subjected to various ranges of stress, unless the complete loading schedule is known. Since this generally is not possible, the best that can be done for increasing the safety of a given steel structure is to apply a large number of cycles of stress below the fatigue limit after each period of overstress. This usually is taken care of by the nature of the service, but in some applications involving exceptionally high stresses it might be worthwhile to apply low stresses purposely to counteract the effect of unusually long periods of high stress.

The results of the limited number of tests made with specimens improved by understressing suggest that this improvement can be measured by distance along a strength-loss line in a manner similar to that used for measuring damage (Fig. 20). If this can be done, then the results of any loading schedule can be predicted, regardless of whether the stresses are above or below the fatigue limit.

CONCLUSIONS

The results given in this paper lead to several important conclusions regarding fatigue damage occurring *prior* to the start of cracking.

1. If the damage occurring at one stress was determined by measurement at

another, the apparent rate of damage depended on the value of both stresses. When the first was larger than the second, the damage occurred rapidly at first, then more slowly: the reverse was true when the first stress was the smaller.

2. The *S-N* curves for specimens after various damaging treatments showed that with increasing damage, the absolute value of the slope of the falling part of the curves increased and the fatigue limit decreased.

3. These differences in the *S-N* curves provided a measure of damage with the help of which the damage occurring at different stresses could be directly added. The performance of specimens tested under complex loading schedules could be ascertained by the use of cycle-ratio *versus* damage curves for the stresses involved.

4. The improvement due to understressing apparently can be measured in the same way as damage due to overstress.

5. Fatigue damage prior to the start of cracking was repaired by understressing but not by heat treatment.

Certain other conclusions regarding fatigue phenomena *in general* may be drawn from the results obtained.

6. The slopes of the falling part of the *S-N* curves for the same material in smooth, notched, and cracked specimens showed approximately the same stress-concentration ratios as the fatigue limits.

7. The number of cracks formed in a specimen before fracture increased rapidly as the test stress was raised above the fatigue limit.

8. The length, L, of fatigue cracks may be represented by the equation

$$\log (L - C) = \alpha N$$

where C and α are constants, N is number of cycles.

9. In specimens stressed considerably above the fatigue limit, the number of cracks, M, in a narrow circumferential band of the specimen can be expressed by the equation

$$\sqrt{M} = KS$$

where K is a constant and S is the average stress in the band.

10. The development of a small crack in a specimen resulted in a large decrease in the value of the fatigue limit as compared with specimens which had been stressed just short of cracking.

REFERENCES

(1) J. A. Bennett, "Effect of Fatigue Stressing Short of Failure on Some Typical Aircraft Metals," Nat. Advisory Com. Aeronautics, Technical Note 992, October, 1945.

(2) J. B. Kommers, "The Effect of Overstress in Fatigue on the Endurance Life of Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 532 (1945).

(3) J. A. Bennett, "The Early Detection of Cracks Resulting from Fatigue Stressing," Nat. Advisory Com. Aeronautics, Restricted Bulletin 4I15, September, 1944.

(4) J. O. Almen, "The Useful Data to be Derived from Fatigue Tests," *Metal Progress*, Vol. 44, p. 254 (1943).

(5) R. J. Roark, "Formulas for Stress and Strain," McGraw-Hill, New York, N. Y. (1943).

(6) M. A. Miner, "Cumulative Damage in Fatigue," *Journal Applied Mechanics*, **Vol.** 12, p. A159, September, 1945.

DISCUSSION

Chairman R. E. Peterson.[1]—I was particularly impressed with Mr. Bennett's handling of the data from the statistical viewpoint. Also, I was impressed with the large number of cracks that were found in an ordinary, plain specimen. In some cases there were several hundred cracks, where we ordinarily think of just one crack in that type of specimen. I should like to ask if work was done with other materials and if a similar situation was found with regard to the number of cracks.

Mr. C. A. Adams.[2]—This paper might be criticized along the lines of Mr. Almen's discussion of the Jensen and Moore paper because of the lack of connection between the laboratory conditions and actual operating conditions. On the other hand, my personal attitude is that the paper makes a contribution in that it points out the effect of certain variables which had not been determined with any accuracy before.

Every experimental investigation is a step in adding to our knowledge of the effect of the variables involved. It just happens that I am primarily an electrical engineer, and in that field the application of what some people scornfully refer to as theory can be much more accurately applied than in this very complex field of the behavior of metals under the endless number of combinations of stresses—static, impact, fatigue, etc.

In the electrical field, it is possible to sit down with pencil and paper and a thorough knowledge of the fundamental principles involved, and to design an electrical machine which will do what you want it to do, and to know that it will do it. The only difference between that situation and this one is the complexity of the phenomena and our lack of knowledge of the fundamental phases of the problem. What do we know about the ultimate nature of these phenomena?

We are digging in a little deeper here and there, with the latest scientific devices at our disposal, but we are still a long way from that fundamental knowledge which would enable us to make quantitative predictions as to what would happen under new conditions or with new combinations of variables.

My position, as contrasted to Mr. Almen's, is merely this: Not so much that these results are of little value because they do not simulate the operating conditions, or that we have not yet dug down deep enough into the fundamentals to be able to apply them quantitatively, but that every new experiment when properly interpreted adds something to our understanding of the fundamental nature of these complex phenomena.

Mr. J. A. Bennett (*author*).—In regard to Mr. Peterson's question about other materials, I believe I forgot to call attention to the fact that all of the tests were run on normalized X4130. We have not done this type of thing on any other materials, although we are planning similar tests on aluminum alloys.

In regard to Mr. Adams' remarks with reference to lack of connection to service conditions, we were trying to bridge the gap between laboratory tests and service conditions by making the loading schedules increasingly complicated. That is my justification.

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[1] Manager, Mechanics Div., Westinghouse Research Labs., Westinghouse Electric Corp., East Pittsburgh, Pa.

[2] Consulting Engineer, The Budd Co., Philadelphia, Pa.

The improvement caused by small amounts of overstressing is a subject to which we have given a good deal of thought. This improvement has been reported by several investigators, both for laboratory specimens and machine elements, but we did not find it in this investigation. Such an improvement would drop the first part of the curve of Fig. 21 below the *X*-axis, but I believe the damage would still be additive.

In the future we hope to extend the work to improved specimens. The one point determined in this investigation indicated that improvement could be handled by the same methods used for damage, and if that turns out to be correct, we should be able to predict the life of a laboratory specimen under any program of loading.

Certainly, then, we have moved a step closer to the service conditions, where the stress is fluctuating in a random manner.

MR. J. B. KOMMERS (*by letter*).[3]—This paper was especially interesting to me because for the past ten years I have been studying the changes that occur in fatigue specimens when subjected to stresses either above or below the ordinary endurance limit. The primary object of my experiments was to determine the changes that occur both in endurance limit and endurance life while the specimen is being subjected to fatigue stresses.

In his opening paragraph the author makes the statement that overstress does not change the usual mechanical properties of the material. In my paper, presented before the Society in 1938,[4] I quoted some results by Memmler and Laute which showed that overstress in fatigue can change the yield point, elastic limit, and percentage of elongation.

It has been assumed by some writers that when, say, 50 per cent of the endurance life has been used up at one overstress, and if the overstress is then increased or decreased, that the same 50 per cent decrease in endurance life will carry over at the new overstress. This may occasionally be true for some stresses and some materials, but it is not generally true as shown by Fig. 7 of the author's paper and the results given in my 1945 A.S.T.M. paper.[5] If such carry-over of damage to endurance life were a correct assumption, the experimental points in Fig. 7 should fall on the dashed 45-deg. line.

Another point brought out by Fig. 7, which is similar to results that I have found, is that when the first overstress is high and the second lower, the damage is greater than the equal damage represented by the 45-deg. line, and when the first overstress is low and the second higher, the damage is less than equal damage.

I wish to congratulate the author on the ingenuity he has shown in constructing Fig. 21, by means of which the damage may be predicted for a series of different overstresses. If a greater variety of prestresses were used, a family of curves could be developed which would augment the usefulness of the diagram. Even if such results are only approximate, they represent an advance in technique.

In the past, various methods have been tried in an attempt to repair the damage to endurance limit caused by overstress. So far as I am aware, this can be accomplished only by understressing or a combination of understressing and coaxing. In 1937,[6] I reported results similar to the author's on the effect of understressing in repairing damage.

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[3] Professor of Mechanics, University of Wisconsin, Madison, Wis.

[4] J. B. Kommers, "The Effect of Overstressing and Understressing in Fatigue," Am. Soc. Testing Mats., Vol. 38, Part II, p. 249 (1938).

[5] J. B. Kommers, "The Effect of Overstress in Fatigue on the Endurance Life of Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 532 (1945).

From my experiments I have concluded that the number of cycles after a crack first developed was small, while the author's experiments with his particular steel show that this is not always the case. His method of setting the testing machine to shut off at a fixed deflection of the specimen should prove to be a generally useful device.

MR. BENNETT.—Mr. Kommers has called attention to an inaccurate statement in the first paragraph of the paper. During fatigue stressing the metal is being cold worked, and the mechanical properties will change accordingly. However, it is not possible to distinguish between cold work which is damaging and that which is beneficial; that is there is no change in mechanical properties corresponding to the change in fatigue properties.

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[6] J. B. Kommers, "Overstressing and Understressing in Fatigue," *Engineering*, (London), Vol. 143, p. 620 (1937).

THE NOTCH SENSITIVITY IN FATIGUE LOADING OF SOME MAGNESIUM-BASE AND ALUMINUM-BASE ALLOYS*

By George H. Found[1]

Synopsis

Fatigue tests of the rotating-beam, axial loading, and plate bending types were performed on a number of commercial magnesium and aluminum-base alloys. The results are presented, but they are not believed to be directly applicable for quantitative design purposes. They are used, however, to draw general conclusions regarding the use of magnesium and aluminum under various conditions of stress concentration, temperature, and required life.

The nature of the structural applications for which the light metal alloys are generally used is such that their service performance is frequently governed by their fatigue properties. For this reason, increasing regard is being paid to the fatigue characteristics of these materials.

Closely associated with fatigue properties is notch sensitivity, or the effect of concentrated polyaxial stresses on metal strength. Experience shows that service failures usually occur at stress concentrations which are inevitably associated with methods of joining parts, with general design, and with conditions of surface finish. Service experience with magnesium castings has shown that in highly stressed regions the stress-concentration factor associated with design should be maintained below 1.3. In the vicinity of screw threads and bolt and stud holes, severe stress concentrations may exist when service load is applied. In such regions, where the stress-concentration factor may be as high as 5, the nominal stress is maintained at a low level by the use of thicker sections and bosses. In wrought metal structures where stress concentrations as high as 9 may exist at joints, various expedients for reinforcement, such as the use of butt straps and gussets, are resorted to in order to reduce the nominal stress.

Since the importance of stress concentrations is thus quite evident, this paper includes data from notched-bar fatigue tests as well as from plain-bar fatigue tests. The former provide a measure of the notch sensitivity in fatigue of the various materials investigated.

The most common sources of fatigue information are the results obtained from tests run on several types of laboratory fatigue testing machines. Polished test bars of standardized design and surface finish are generally used. The fatigue data presented herein are intended to provide a basis for qualitative comparison of alloys under various types of load application and various condi-

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946

[1] The Dow Chemical Co., Midland, Mich.

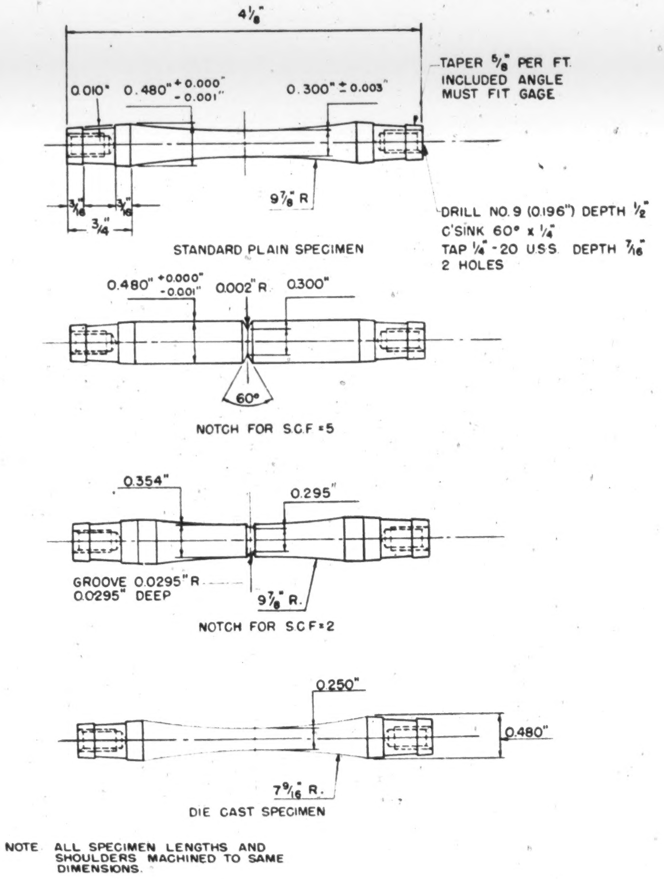
4⅛"
TAPER ⅝" PER FT.
INCLUDED ANGLE
MUST FIT GAGE
0.010"
0.480" +0.000" -0.001"
0.300" ± 0.003"
9⅞" R
3/16"
3/16"
¾"
DRILL NO. 9 (0.196") DEPTH ½"
C'SINK 60° x ¼"
TAP ¼" - 20 U.S.S. DEPTH 7/16"
2 HOLES
STANDARD PLAIN SPECIMEN
0.480" +0.000" -0.001"
0.002" R
0.300"
60°
NOTCH FOR S.C.F = 5
0.354"
0.295"
GROOVE 0.0295" R
0.0295" DEEP
9⅞" R.
NOTCH FOR S.C.F = 2
0.250"
0.480"
7 9/16" R.
DIE CAST SPECIMEN
NOTE ALL SPECIMEN LENGTHS AND SHOULDERS MACHINED TO SAME DIMENSIONS.

FIG. 1.—Dimensions of the Specimens Used for **Rotating-Beam Fatigue Tests.**

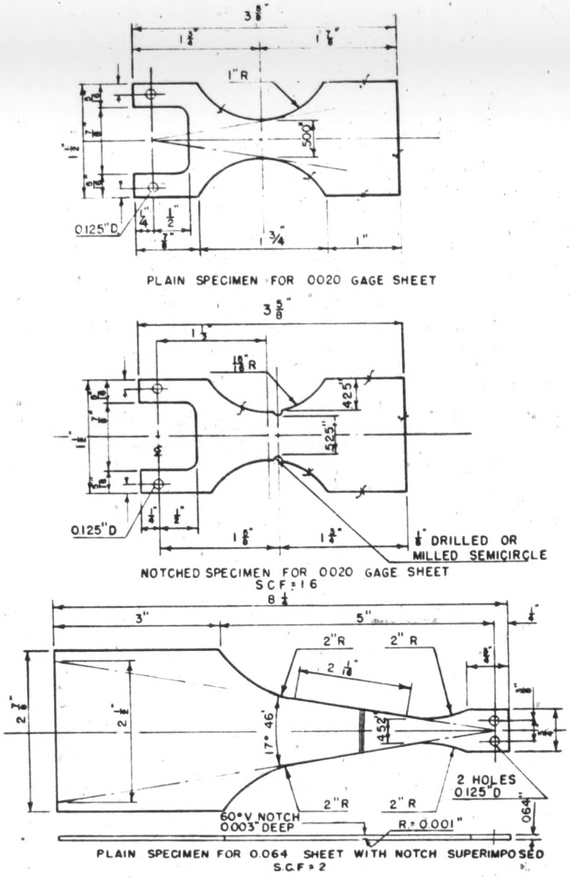
3⅝"
1"R
.500"
0.125"D
1¾"
1"
PLAIN SPECIMEN FOR 0020 GAGE SHEET
3⅝"
R
.425"
.525"
0.125"D
⅛" DRILLED OR MILLED SEMICIRCLE
NOTCHED SPECIMEN FOR 0020 GAGE SHEET
S.C.F = 1.6
8¼"
3"
5"
2"R
2"R
2"R
2"R
17° 46'
.452"
2 HOLES
0.125" D
.064"
60° V NOTCH
0.003" DEEP
R = 0.001"
PLAIN SPECIMEN FOR 0.064 SHEET WITH NOTCH SUPERIMPOSED
S.C.F = 2

FIG. 2.—Dimensions of the Specimens Used for Plate Bending Fatigue Tests.

tions of temperature, desired life range, and notching.

It is important to note that the results from these fatigue-bar tests on magnesium and aluminum alloys have been found to be of but limited use for predicting actual stress ranges for safe dynamic loading of parts and assemblies in service. In the case of castings, this can be associated with the metallurgical differences between test bars and actual parts; and also with the relative effects of surface finish on test-bar fatigue properties as compared to the effects on parts. Often for wrought assemblies, which incorporate sheet, extrusions or forgings, the strength of joints is the determinative fatigue property of structures, while the fatigue properties of the unnotched metal are of secondary importance. Further, in the case of both cast and wrought metal, the dissimilarity in the way in which load is applied to fatigue bar specimens as compared to full-scale parts under service loading accounts for an additional discrepancy between the fatigue properties of test bars and actual parts. How each of the above variables affects the results is beyond the scope of the present paper. Suffice it to say that it is recommended that the test-bar data presented here not be used quantitatively for design values, but rather, simply for a qualitative comparison of materials.

TYPES OF FATIGUE TESTING

The types of fatigue tests for which data are given are the rotating beam in pure bending, the cantilever plate bending, and the axial or direct loading tests. The various fatigue testing machines in which each of these three types of loading was accomplished are respectively, the standard R. R. Moore, the Krouse flexure plate, and the Krouse direct-loading machines, all of which are well known. The shape and dimensions of the test specimens used in each of these tests are shown in Figs. 1, 2, and 3.

A summary of the salient features of each of these three fatigue bar tests is of interest for full evaluation of the testing:

Tests for room-temperature data were made on standard R. R. Moore machines, some of which operated at 10,000 and others at 3000 rpm. This difference in rate of stressing did not affect the results. Due to unavoidable heating effects in the bearings, the temperature of the specimens during test was about 100 F. However, the tests were insensitive to this deviation from normal room temperature.

Tests at 250 and 300 F. were also run on the standard R. R. Moore machines. A small tube furnace surrounding each specimen, with clearance just adequate to permit proper machine operation was satisfactory for this testing. The temperature at a point $\frac{1}{16}$ in. from the bar was calibrated against the bar temperature when the fatigue machine was not running. The change in temperature when the bar was set in motion was determined by making the measurements immediately after the bar had run at equilibrium temperature for several hours. The specimen temperatures were judged to be accurate to within ±5 F. Excessive heating of the bearings in the housings of the fatigue machines occurred due to thermal conduction through the specimens into the specimen collets. This was reduced by the use of ceramic collet inserts. Incompleted tests at 400 F. have demonstrated that further modifications of this system are necessary if temperatures in excess of 400 F. are to be obtained in the standard R. R. Moore tests.

The flexural (plate-bending) fatigue

test data presented were obtained at room temperature only. For these tests the load was applied by an eccentrically driven loading arm attached to the outer end of the cantilever-beam specimens. Sheet, cast panels, and flat extrusions have been tested in this type of fatigue test. Because the inertia effects of the loading arm were accentuated in relatively long test specimens of thin gage, the smaller specimen size was used for sheet of gage thinner than 0.064 in. These inertia effects were manifest as a whipping action in the motion of the specimen, rather than a uniform bending. This could be seen with the aid of a stroboscope. To reduce further the inertia effects, the loading arms of these machines were made of magnesium alloy. Despite these precautions, additional efforts were necessary when very thin gage sheet (0.020 in. or thinner) was tested at stresses in excess of 15,000 psi. For this purpose an accessory jig was devised, which is shown in Fig. 4. The inertia effects of the loading arm are resisted by this jig. The specimen, which was loaded through the jig, was thus not subjected directly to the inertia effects. The stress at which the specimens operate in this type of testing machine was

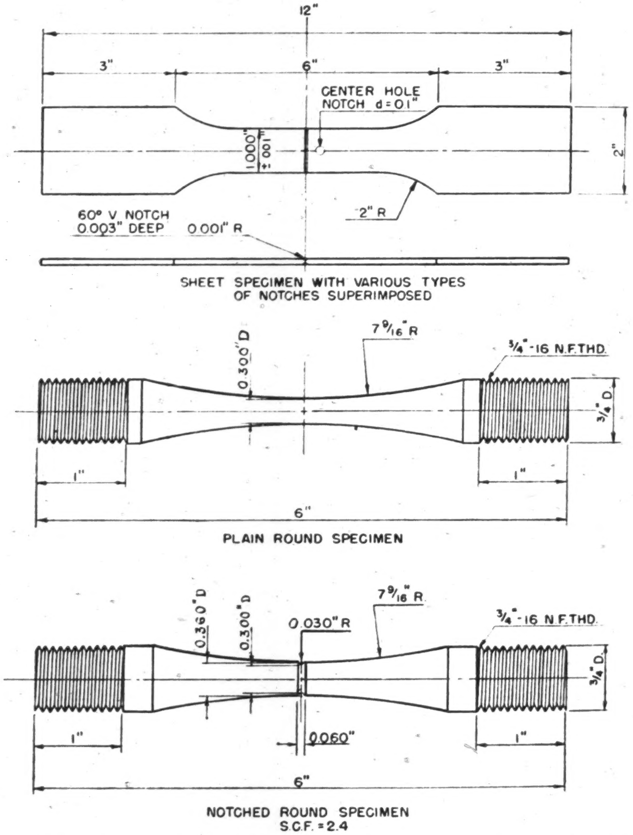
12"
3"
6"
3"
CENTER HOLE
NOTCH d = 0.1"
1.000" ± .001
2"
2" R
60° V NOTCH
0.003" DEEP
0.001" R
SHEET SPECIMEN WITH VARIOUS TYPES
OF NOTCHES SUPERIMPOSED
0.300" D
7 9/16" R
3/4"-16 N.F.THD.
3/4" D
1"
1"
6"
PLAIN ROUND SPECIMEN
0.360" D
0.300" D
0.030" R
7 9/16" R
3/4"-16 N.F.THD.
3/4" D
1"
0.060"
1"
6"
NOTCHED ROUND SPECIMEN
S.C.F. = 2.4

FIG. 3.—Dimensions of the Specimens Used for Axial Loading Fatigue Tests.

calculated on the basis of the dimensions of the specimens and the load necessary to effect the deflections at which the specimens are run. Since such beam calculations were based upon perfect elasticity, at the high stress end of the S-N fatigue curve, the stress often exceeded the elastic limit. For this reason, there was a small inherent inaccuracy for high stress calculations and the actual stress tended to be lower than the calculated stress. This error was less than 2000 psi.

The axial-loading fatigue machines were operated at room temperature and at 275 F. The loading, in the type of machine used, was applied by a lever arm that was connected through a plate fulcra system to the specimen and the lever was flexed by a rotating eccentric. Considerable care had to be exercised in order to obtain grip alignment and,

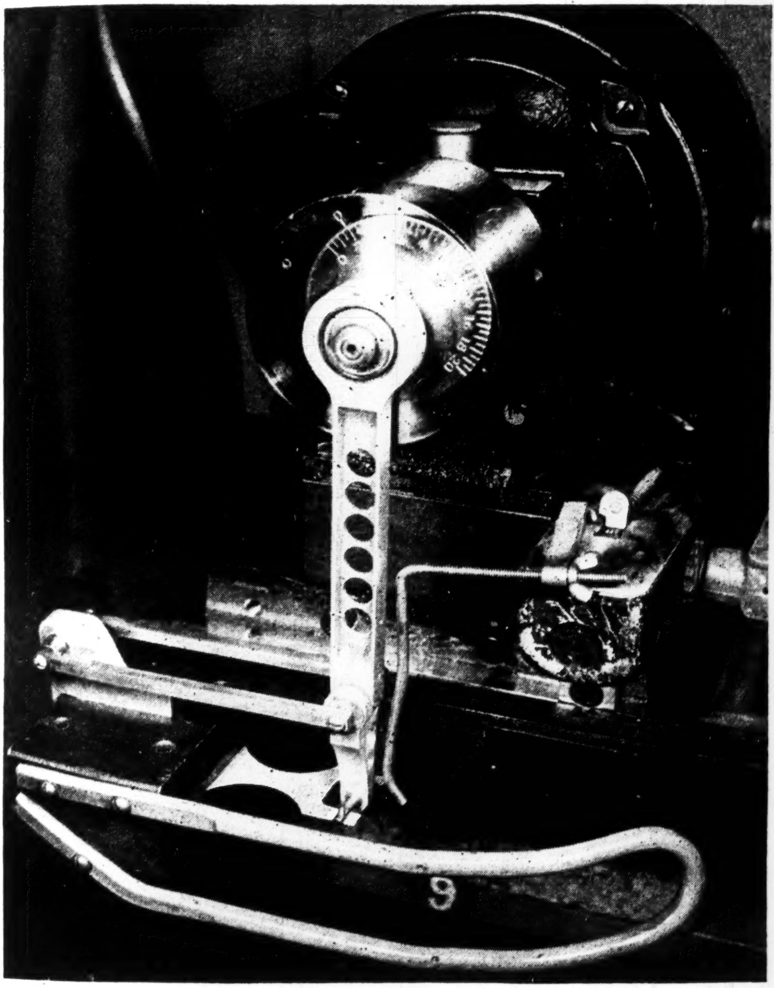

FIG. 4.—Accessory Jig Used on Plate Bending Tests to Reduce Inertia Forces in Thin Sheet.

hence, axial loading. For the tests on sheet, this was accomplished by mechanical methods to within 0.01 in. in 12 in. For the tests on rounds, mechanical methods were again used for aligning the grips. Subsequent checks with electric strain gages mounted on four sides of a cylindrical specimen revealed that the worst case of nonaxiality to be found in any set of grips effected a ±7 per cent variation in stress level across a specimen at 1500 psi. This decreased with increasing applied loads. A major part of the scatter in the axial fatigue test results thus could be attributed to this unavoidable variation in alignment.

TABLE I.—DESIGNATIONS AND NOMINAL COMPOSITIONS OF MATERIALS TESTED.

A.S.T.M. Designation	Dow Designation	Composition, per cent		
		Aluminum	Magnesium	Zinc
	MAGNESIUM			
AZ92	C	9.0	0.1	2.0
AZ63	H	6.0	0.2	3.0
AZ91	R	9.0	0.2	0.6
MZ	M	...	1.5	...
AZ31X	FS-1	3.0	0.3	1.0
AZ61X	J-1	6.5	0.3	0.7
AZ80X	O-1	8.5	0.3	0.5
AZ51X	JS-1	4.8	0.3	0.8

ALUMINUM

Commercial Designation	Chemical Composition, per cent					
	Copper	Magnesium	Manganese	Iron	Nickel	Silicon
195	4.0	...	...	...	...	...
355	1.3	0.5	...	...	...	5.0
356	...	0.3	...	...	...	7.0
122	10.0	0.2	...	1.2	...	...
142	4.0	1.5	...	...	2.0	...
220	...	10.0	...	...	...	...
24S	4.5	1.5	0.6	...	...	...
18S	4.0	0.5	...	...	2.0	...
75S	...	...	...	...	...	...
R-301	4.5	0.5	0.8	<1	...	0.8
13	...	...	...	...	...	12

The type of axial loading fatigue testing machine used maintained a constant deflection during the course of any one test. Relaxation during testing due to plastic deformation in the specimens, however, resulted in a reduction of the stress in the specimen. Periodic readjustments in the loading was, therefore, made during the course of each test. In some machines this was accomplished automatically by built-in compensating equipment. For the current testing, the original load, as well as the corrections, was determined with the aid of dynamic electric strain gages and electric strain analyzing apparatus. The strain gages were fixed to the plate fulcra of the testing machines. The necessary corrections were made manually. The correcting of the loading could be done without stopping the testing machine or otherwise interrupting the test. Load checks were made every 5 min. for the first hour and every 15 min. thereafter until either relaxation ceased or the specimen broke. Usually 2 to 4 hr. were required for the specimens to adjust themselves to resist further relaxation.

The axial-loading fatigue tests were run in the same manner at elevated temperature as at room temperature, except for more frequent and more extended checks of the relaxation. Tube furnaces were used to enclose the specimens. The temperature was maintained to ±5 F.

MATERIAL TESTED

Both cast and wrought magnesium-base and aluminum-base alloys were tested. These materials were of commercial quality and were of acceptable mechanical properties.[2] The alloys are listed and their nominal compositions and typical mechanical properties are given in Tables I and II, respectively.

The sand-cast magnesium specimens used were free from porosity and were of average grain diameter between

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[2] See Note *b*, Table II.

0.003 and about 0.008 in. The sand and permanent mold cast aluminum test bars were made and heat-treated by a commercial aluminum foundry. The wrought metal specimens were cut from batches of commercial sheet and extrusions. The die-cast bars were cast to shape according to normal commercial practice. The specimens of forged material were cut from forged stock which was also prepared according to commercial practice.

RESULTS OF TESTS

The shape of the fatigue curves varies for aluminum- and magnesium-base alloys as is demonstrated in the sample curves in Figs. 5, 8, 9, and 10. The curves for the magnesium-base alloys frequently flattened out in the vicinity of 10^6 to 10^7 cycles for the tests involving bending. In the axial-loading fatigue tests the flattening sometimes occurred at less than 10^6 cycles, particularly in the case of sheet alloys. The curves for aluminum rarely flattened out at less than 10^8 cycles. In general, by virtue of this tendency for flatness in the long-life portion of the curve, the magnesium-base alloys gain either equality with, or superiority over, the aluminum alloys at long life. In addition, the short-life portion of the fatigue curves for aluminum often has a significantly steeper slope than do the curves for magnesium. This was particularly true in bending fatigue tests. The terms "short life" and "long life" are used throughout to differentiate between the portions of the fatigue curve of the characteristics just described. These regions are around 10^5 cycles, and 10^7 cycles and greater, respectively.

Rotating Beam Tests:

Test bars were prepared according to a given machining and polishing schedule. The final machining cuts were reduced to 0.010 in. to minimize the depth of cold-working at the surface. The notched bars were then tested without further preparation. The plain bars were polished with a series of abrasive papers starting with No. 320 and ending with a No. 000 metallographic abrasive paper. To complete the polishing, suspensions of alundum

TABLE II.—TYPICAL MECHANICAL PROPERTIES OF MATERIALS TESTED.

Alloy and Heat Treatment	Tensile Strength, psi.	Yield Strength, psi.	Elongation in 2 in., per cent	Compression Yield Strength, psi.
SAND CASTING ALLOYS				
C-AC	24 000	14 000	2	.....
C-HT	40 000	16 000	10	.....
C-HTS	40 000	20 000	3	.....
C-HTA	40 000	23 000	2	.....
H-AC	29 000	14 000	6	.....
H-ACS	29 000	14 000	5	.....
H-HT	40 000	14 000	12	.....
H-HTS	40 000	17 000	7	.....
H-HTA	40 000	19 000	5	.....
195-T6	36 000	24 000	5.0	.....
355-T6	35 000	25 000	2.5	.....
356-T6	33 000	24 000	4.0	.....
122-T2	27 000	20 000	0.5	.....
142-T61[a]	37 000	32 000	<0.5	.....
220-T4	46 000	25 000	14.0	.....
DIE CASTINGS				
R	33 000	22 000	3	.....
Alcoa No. 13	37 000	18 000	1.8	.....
EXTRUSIONS				
M	38 000	26 000	10	.....
FS-1	40 000	30 000	15	.....
J-1	45 000	32 000	15	.....
O-1HTA	50 000	34 000	7	.....
24S-T	68 000	46 000	22	.....
18S-T[b]	52 000	41 000	8.5	.....
FORGINGS				
J-1	43 000	26 000	12	.....
O-1	46 000	31 000	8	.....
O-1A	50 000	34 000	6	.....
O-1HTA	50 000	34 000	5	.....
SHEET				
J-1a	43 000	26 000	16	16 000
J-1h	47 000	34 000	9	27 000
FS-1a	37 000	22 000	21	16 000
FS-1h	43 000	33 000	11	26 000
Ma	33 000	15 000	17	12 000
Mh	37 000	29 000	8	20 000
JS-1a	41 000	22 000	18	15 000
JS-1h	44 000	33 000	9	27 000
24S-T	68 000	46 000	19	.....
75S-T	76 000	66 000	11	.....
R301-T	68 000	61 000	9	.....

[a] Cast by permanent mold methods.

[b] Rolled and straightened rod. 950 F., 12 hr., *Q.*, 450 F. 16 hr.—Batch properties not, typical properties.

and then levigated alumina followed by dry buffing were used. This procedure conferred a specular surface finish and removed the 0.001 to 0.002-in. surface layer containing the most heavily worked metal. In this test the mean stress during the testing was zero.

Fatigue curves from several rotating-beam tests are shown in Fig. 5. In order systematically to construct the fatigue curves on the basis of test points which have the usual scatter characteristics, the method of least squares was used in calculating the slope and intercepts of the fatigue curves. This was facilitated by the fact that the stress-life relationship for the test points was linear when plotted on logarithmic coordinates. Data from rotating-beam tests on plain and notched specimens at room and elevated temperatures appear in Table III. These results are for both magnesium and aluminum alloys in both cast and wrought form, the tests curves being run out to 10^8 cycles. Since in most cases the fatigue curves for magnesium alloys flatten at about 10^7 cycles, running specimens beyond 10^8 cycles was unnecessary.

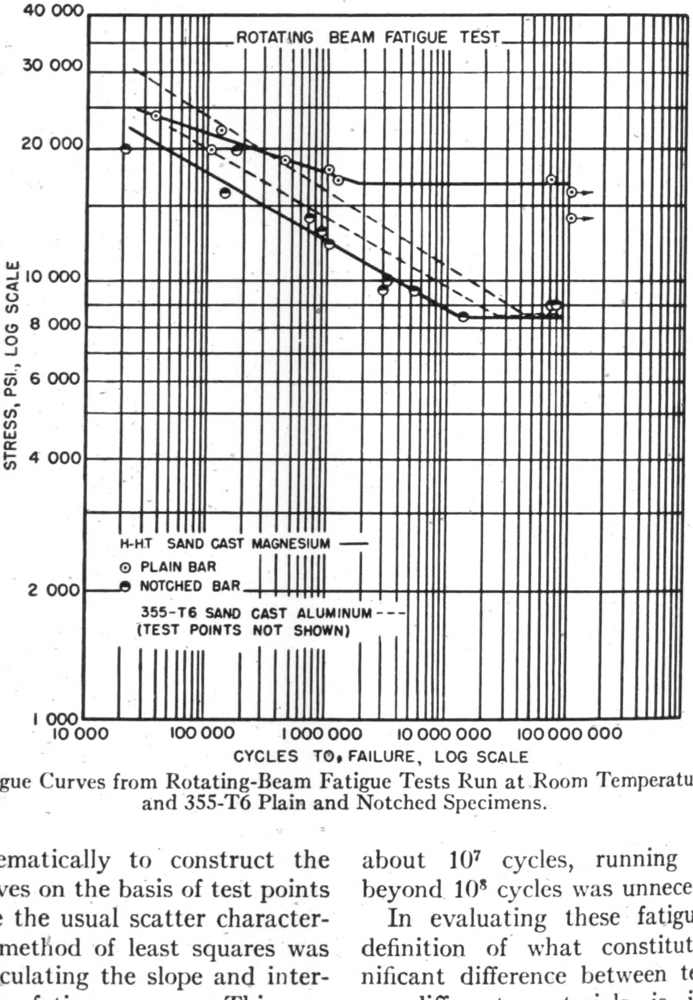

FIG. 5.—Fatigue Curves from Rotating-Beam Fatigue Tests Run at Room Temperature on H-HT and 355-T6 Plain and Notched Specimens.

In evaluating these fatigue data a definition of what constitutes a significant difference between test results on different materials is important. By statistical analysis, the standard error was calculated for the rotating-beam tests. At stress levels in excess of 10,000 psi., the possible error due to testing and also to unintentional batch differences was about ±1000 psi. Below 10,000 psi. the possible error was about ±500 psi. Thus, differences between curves, to be significant, must be at least 2000 psi. at stresses above 10,000 and 1000 psi. below 10,000 psi.

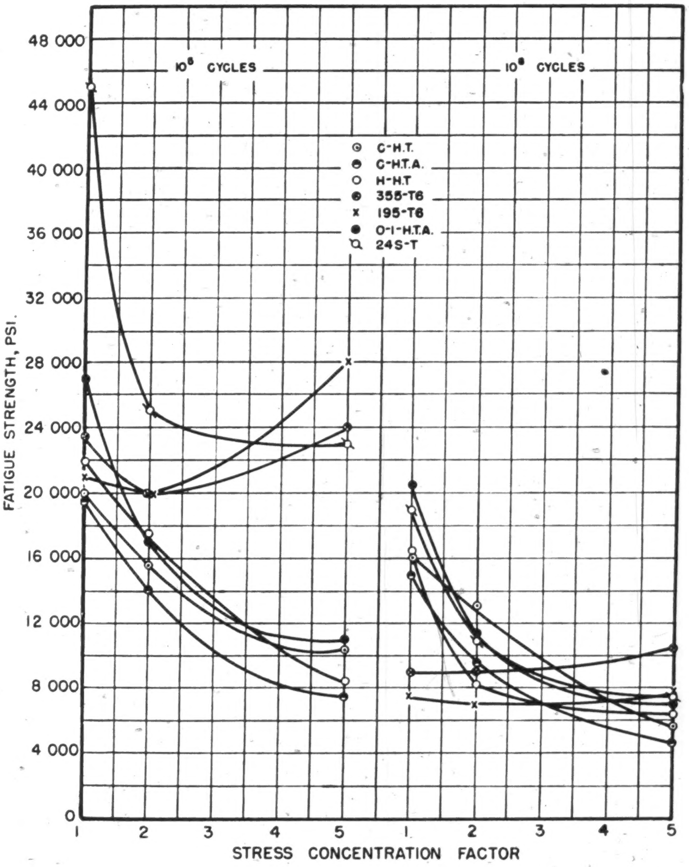

FIG. 6.—Data from Rotating Beam Fatigue Tests Showing the Effect of Notches of Various Stress-Concentration Factors on the Fatigue Properties of Several Magnesium and Aluminum Alloys.

On this basis, data for plain bars in the rotating-beam tests in Table III may be evaluated. Of the magnesium C and H sand-casting alloys, the HT, HTS, and HTA states are in the same class of superiority at both short and long life at room temperature. The cast aluminum alloys are of equal or slightly superior dynamic strength for short-life application, but appear to be, at best, about two thirds the cast magnesium test bar fatigue strength for extended life.

In Fig. 6 are the results of tests on notched bars of several alloys, taken from Table III and plotted as a function of degree of notching. The results (S.C.F.[3] = 2) show the cast aluminum to be

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[3] S.C.F. = Stress-concentration factor as determined from geometry of notch and nature of applied loading.

TABLE III.—ROTATING BEAM FATIGUE DATA AS A FUNCTION OF TEMPERATURE AND NOTCHING.

Alloy and Heat Treatment	Plain Bar Properties, psi.			Notched Bar Properties, Stress Concentration Factor = 2, psi.			Notched Bar Properties, Stress Concentration Factor = 5, psi.		
	10^5[a]	10^7	10^8	10^5	10^7	10^8	10^5	10^7	10^8
SAND CASTINGS ROOM TEMPERATURE									
C-AC	18 500	14 500	13 000	11 000	8 500	7 500	8 500	5 000	4 000
C-HT	20 000	16 500	16 000	15 500	13 500	12 500	10 000	6 000	5 500
C-HTS	20 500	15 000	13 000	14 500	10 000	9 000	8 000	6 500	6 000
C-HTA	19 500	16 000	15 000	14 000	9 500	9 500	7 500	5 000	4 500
H-AC	18 000	10 500	10 500	13 500	7 000	7 000	8 500	5 000	4 000
H-ACS	23 000	15 000	12 000	12 500	9 500	8 500	.....	.....	.....
H-HT	21 500	16 500	16 500	17 500	9 000	8 500	8 500	7 000	6 500
H-HTS	21 000	16 000	15 000	14 000	10 500	9 500	7 500	6 000	5 500
H-HTA	18 000	16 000	15 000	11 500	9 500	9 000	8 500	5 000	3 500
195-T6	21 000	10 000	7 500	20 000	7 500	7 000	28 000	11 500	7 500
355-T6	23 500	11 000	8 500	20 000	9 500	8 500	24 000	13 500	10 500
356-T6	22 000	11 500	10 000	19 000	10 500	8 500	.....	.....	.....
122-T2	17 000	10 500	10 000	11 000	7 000	5 500	.....	.....	.....
220-T4	19 500	10 500	8 000	17 000	9 000	6 500	.....	.....	.....
250 F.									
C-HTA	14 500	11 500	10 500	13 500	8 000	6 000	.....	.....	.....
H-HTA	17 000	12 500	11 000	11 500	7 000	7 000	.....	.....	.....
300 F.									
C-HTS	15 500	10 000	9 000	11 000	5 500	4 500	.....	.....	.....
C-HTA	16 000	11 000	10 000	11 500	6 500	6 000	.....	.....	.....
H-HTS	13 500	9 500	9 500	10 000	5 500	5 000	.....	.....	.....
H-HTA	.....	9 000	7 500	9 500	6 000	5 000	.....	.....	.....
195-T6	21 000	9 000	9 000	17 500	7 500	7 000	.....	.....	.....
355-T6	20 500	11 000	10 000	19 500	8 000	8 000	.....	.....	.....
356-T6	20 500	10 000	10 000	17 000	8 500	8 000	.....	.....	.....
122-T2	14 500	9 500	8 000	13 000	7 500	7 000	.....	.....	.....
PERMANENT MOLD CASTINGS ROOM TEMPERATURE									
C-HTA	20 500	16 000	14 000	16 000	10 500	8 500	.....	.....	.....
142-T6	20 000	10 500	9 000	15 000	8 000	8 000	.....	.....	.....
300 F.									
142-T6	18 000	10 000	9 500	15 000	7 500	7 500	.....	.....	.....
DIE CASTINGS ROOM TEMPERATURE									
R (c)	17 500	15 000	15 000	.....	.....	.....	.....	.....	.....
Alcoa No. 13[b]	16 000	9 500	9 500	.....	.....	.....	.....	.....	.....
EXTRUSIONS ROOM TEMPERATURE									
M	15 500	10 500	10 500	11 000	7 000	5 500	.....	.....	.....
FS-1	27 000	21 000	19 000	16 000	10 500	9 000	.....	.....	.....
J-1	24 500	20 500	20 000	16 000	11 500	10 000	11 500	6 500	6 000
O-1	27 000	22 500	20 500	16 500	12 500	11 000	10 500	7 000	6 000
O-1HTA	27 000	22 500	20 500	17 000	13 000	11 000	11 000	8 000	7 000
24S-T	45 000	24 000	19 000	25 000	13 000	11 000	23 000	10 500	7 500
18S-T[c]	33 000	22 000	18 000	21 000	11 000	9 000	.....	.....	.....
FORGINGS ROOM TEMPERATURE									
J-1	27 500	21 500	19 500	14 500	11 000	9 500	.....	.....	.....
O-1	28 000	22 000	19 500	16 500	11 500	10 500	.....	.....	.....
O-1A	25 000	20 000	18 000	16 500	9 500	9 500	.....	.....	.....
O-1HTA	26 000	16 000	16 000	14 500	9 500	9 500	.....	.....	.....
300 F.									
O-1A	20 500	11 000	10 500	10 500	6 500	5 000	.....	.....	.....

[a] Number of cycles to failure.
[b] As die cast surfaces.
[c] Rolled rod.

somewhat superior in notched strength at short life to cast magnesium alloys.[4] For long-life applications, the notched-bar strengths of the best aluminum and magnesium casting alloys are about the same, except for C-HT which was superior to all others.

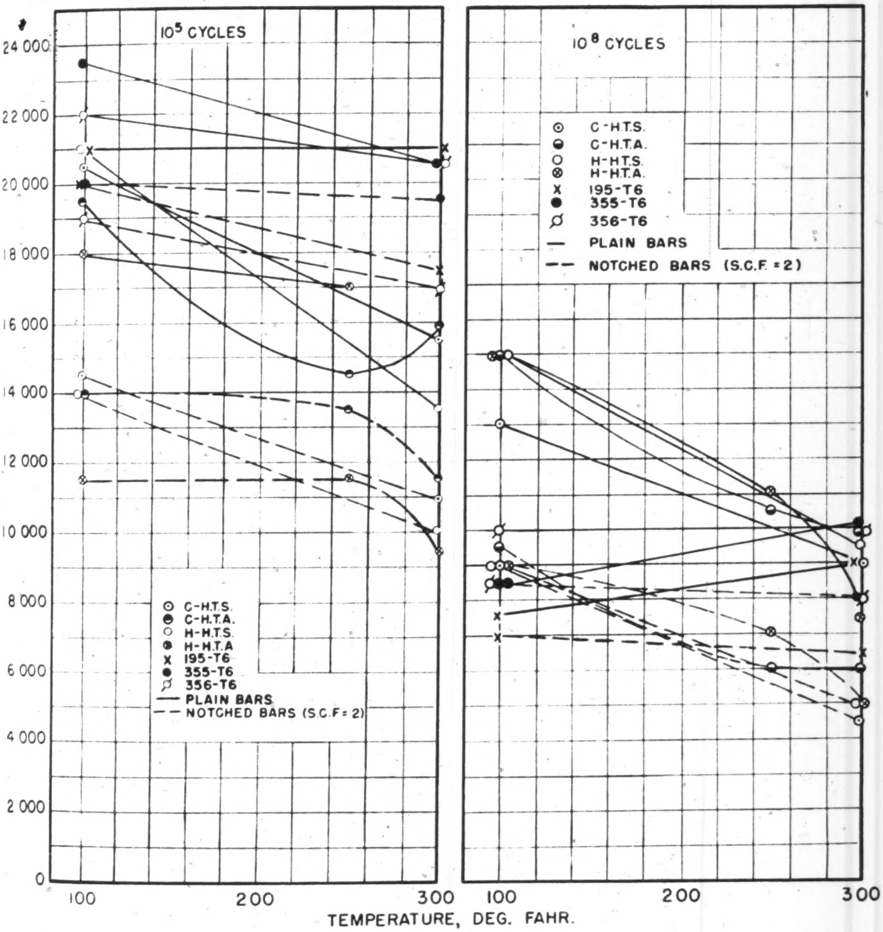

FIG. 7.—Data from Rotating Beam Fatigue Tests Showing the Effect of Elevated Temperature on Several Magnesium and Aluminum Casting Alloys.

The effect on the fatigue bar properties of a sharp notch (S.C.F., approximately 5, see Fig. 1 for geometry)[5] was quite different in magnesium than in aluminum casting alloys, the fatigue properties for magnesium being decreased slightly less than the proportional increase in stress concentration. However, the fatigue properties of cast aluminum increased greatly at short life and slightly at long life. It is apparent that the aluminum casting alloys were less notch sensitive in this fatigue test to severe concentrations of stress than were the magnesium casting alloys.

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[4] R. E. Peterson and A. M. Wahl, "Two and Three Dimensional Cases of Stress-Concentration, and Comparison with Fatigue Tests," *Transactions*, Am. Soc. Mechanical Engrs., March, 1936, Vol. 58, p. A15.

[5] G. N. Neugebauer, "Stress Concentration Factors and Their Effect on Design," *Product Engineer*, February, 1943, Vol. 14, pp. 82, 168.

Data for rotating-beam tests at elevated temperatures from Table III are plotted in Fig. 7. At short life the highest fatigue properties for the plain bars of aluminum casting alloys were superior to those of magnesium casting alloys but at long life there was no appreciable difference. The properties of notched bars of aluminum were generally superior to C and H magnesium casting alloys under any condition at elevated temperature.

Plain bars of two permanent mold casting alloys representative of magnesium and aluminum, C-HTA and 142-T6, had about the same strength at short life. However, for long life, C-HTA had about 50 per cent greater strength than 142-T6. The notched-bar properties (S.C.F. = 2) were similar for the two alloys. With this exception, the comparison between permanent mold casting alloys of the two metals was similar to that between the sand-cast alloys.

TABLE IV.—ROTATING BEAM FATIGUE DATA—NOTCH FACTORS.

Alloy and Heat Treatment	Notch Factors Stress-Concentration Factor = 2 Room Temperature			Notch Factors Stress Concentration Factor = 5 Room Temperature			Notch Factors Stress Concentration Factor = 2 300 F.		
	10^5	10^7	10^8	10^5	10^7	10^8	10^5	10^7	10^8
SAND CASTINGS									
C-AC	0.59	0.59	0.58	0.46	0.35	0.31	...	...	...
C-HT	0.78	0.82	0.78	0.50	0.36	0.29	...	...	...
C-HTS	0.71	0.67	0.69	0.39	0.23	0.22	0.71	0.55	0.50
C-HTA	0.72	0.59	0.63	0.38	0.31	0.30	0.72	0.59	0.60
H-AC	0.75	0.67	0.67	0.47	0.48	0.39	...	...	
H-ACS	0.54	0.63	0.71	...		...	...	...	
H-HT	0.81	0.55	0.51	0.39	0.43	0.39	...	...	...
H-HTS	0.67	0.66	0.63	0.36	0.37	0.37	0.74	0.58	0.53
H-HTA	0.64	0.59	0.60	0.47	0.31	0.23	...	0.67	0.67
195-T6	0.95	0.75	0.93	1.34	1.15	1.00	0.83	0.83	0.78
355-T6	0.85	0.86	1.00	1.02	1.23	1.24	0.95	0.73	0.80
356-T6	0.86	0.91	0.85	...	...	...	0.83	0.85	0.80
122-T2	0.65	0.67	0.55	...	...	...	0.90	0.79	0.88
220-T4	0.87	0.86	0.81	...	...	...	...	...	...
PERMANENT MOLD CASTINGS									
C-HTA	0.78	0.66	0.61	...	...	...	...	...	
142-T6	0.75	0.76	0.89	...	...	...	0.83	0.75	0.79
EXTRUSIONS									
M	0.71	0.67	0.52	...	...	...	...	...	
FS-1	0.59	0.50	0.47	...	...	...	...		
J-1	0.65	0.56	0.50	0.47	0.32	0.30	...		
O-1	0.61	0.56	0.54	0.43	0.31	0.39	...		
O-1HTA	0.63	0.58	0.54	0.41	0.36	0.34	...		
24S-T	0.56	0.54	0.58	0.51	0.44	0.39	...	...	
18S-T	0.64	0.50	0.50	...	...	...	...	...	
FORGINGS									
J-1	0.53	0.51	0.49	...	...	...	...	...	
FS-1	0.80	0.68	0.65	...	...	...	...	...	
O-1	0.57	0.52	0.54	...	...	...	...	...	
O-1A	0.66	0.48	0.53	...	...	...			
O-1HTA	0.56	0.59	0.59	...	...	...	0.51	0.59	0.48

Rotating-beam test bars of magnesium-base R alloy and aluminum-base 13 alloy were die cast (Fig. 1). In order to approximate service conditions they were tested with the die-cast surface as-received. The comparison for plain bars of the two was about the same as that for the sand-cast alloys, the magnesium bars being about 50 per cent stronger at long life and of equal strength to the aluminum alloy at short life.

Test specimens were cut from ½-in. extruded rounds. Magnesium-base M alloy, an alloy of moderate strength, good weldability, and hot formability, had the lowest fatigue properties for both plain and notched bars. All of the other extrusion alloys, FS-1, J-1, O-1, O-1HTA, and 24S-T (including 18S-T ¾-in. rolled rod), for similar specimens had about the same long-life fatigue strength, but for short-life applications, the specimens from the aluminum-base extrusions were superior.

Of the forged magnesium alloys, J-1 and O-1 had the highest strength, plain bars with the fiber direction parallel to the direction of loading being tested.

In Table IV the relative effects of notching on the fatigue properties are expressed in terms of the "notch factor" (notched-bar strength divided by the plain-bar strength). This factor thus provides a measure of the sensitivity to notches. Experimental errors contributed to an estimated accuracy of the notch factor of ±0.1.

TABLE V.—PLATE BENDING FATIGUE DATA AT ROOM TEMPERATURE—MEAN STRESS EQUALS ZERO.

Alloy and Temper	Gage	Plain Bar Properties			Notched Bar Properties[a]			Notch Factors		
		10^6	10^7	10^8	10^6	10^7	10^8	10^6	10^7	10^8
SHEET ALLOYS										
J-1a	0.020	23 500	15 000	14 000	15 000	11 500	11 500[b]	0.64	0.77	0.82
J-1h	.....	30 000	21 000	18 000	15 000	10 500	10 500	0.50	0.50	0.58
FS-1a	.....	19 500	15 500	15 500	17 000	13 000	13 000	0.87	0.84	0.84
FS-1h	.....	20 000	19 500	17 500	19 500	13 000	11 000	0.95	0.62	0.63
Ma	.....	16 000	10 000	10 000	10 000	5 000	5 000	0.63	0.50	0.50
Mh	.....	16 000	10 500	10 500	10 500	5 000	4 500	0.65	0.48	0.43
J-1a	0.064	18 000	14 500	14 000	24 000	15 500[b]	.....	1.33	1.07	...
J-1h	.....	19 000	16 000	15 000	21 000	16 500	.....	1.10	1.03	...
FS-1a	.....	15 000	14 000	13 500	18 000	13 000	.....	1.20	1.08	...
FS-1h	.....	17 000	15 500	15 000	21 500	15 000	.....	1.27	0.97	...
Ma	.....	13 500	9 000	7 500	15 500	9 500	.....	1.15	1.03	...
Mh	.....	16 000	10 500	10 000	15 500	10 000	.....	0.97	1.00	...
JS-1a	.....	19 500	14 500	14 000	19 500	14 500	.....	1.00	1.00	...
JS-1h	.....	24 000	16 000	16 000	22 500	15 000	.....	0.94	0.94	...
24S-T	0.020	43 000	25 500	19 500	37 000	19 000	15 500[b]	0.86	0.80	0.80
75S-T clad	.....	35 000	20 000	19 500	.....	.....	.....	...	...	...
R301-T clad	.....	34 000	20 500	16 000	.....	.....	.....	...	...	...
24S-T	0.064	32 500	19 000	15 000	45 000	22 500[b]	.....	1.38	1.18	...
75S-T clad	.....	26 000	13 500	12 000	34 000	16 500	.....	1.31	1.22	...
R301-T clad	.....	30 500	14 500	14 500	.....	.....	.....	...	...	...
EXTRUSIONS										
M	0.125	14 000	10 000	10 000	.....	.....	.....	...	...	...
FS-1	0.117	17 000	10 000	10 000	.....	.....	.....	...	...	...
J-1	0.114	19 000	13 500	13 500	.....	.....	.....	...	...	...
O-1	0.117	20 000	12 500	12 500	.....	.....	.....	...	...	...
O-1A	0.116	19 500	13 000	13 000	.....	.....	.....	...	...	...
CASTING ALLOYS										
C-HT	0.200	15 000	10 000	10 000	.....	.....	.....	...	...	...
195-T6	0.200	21 000	8 000	.....	.....	.....	.....	...	...	...

[a] Type of notch used for 0.020-in. sheet is the edge notch S.C.F. = 1.6, see Fig. 2. Type of notch used for 0.064-in. sheet is the surface notch, see Fig. 2.

[b] Stress calculated on basis of notched cross-section for both surface notches on 0.064-in. sheet and edge notches on 0.020 in. sheet.

As a class, the aluminum casting alloys, with the exception of 122-T2 which was inferior, had notch factors from 0.75 to 1.00 for S.C.F. = 2, while the notch factors for the magnesium casting alloys averaged from about 0.60 to 0.80. At S.C.F. = 5, the aluminum casting alloys had higher notch factors than the magnesium casting alloys. At 300 F., the notch factors of the aluminum casting alloys (S.C.F. = 2) for all life ranges were between 0.73 and 0.95 and under the same conditions those of magnesium casting alloys were between 0.50 and 0.75.

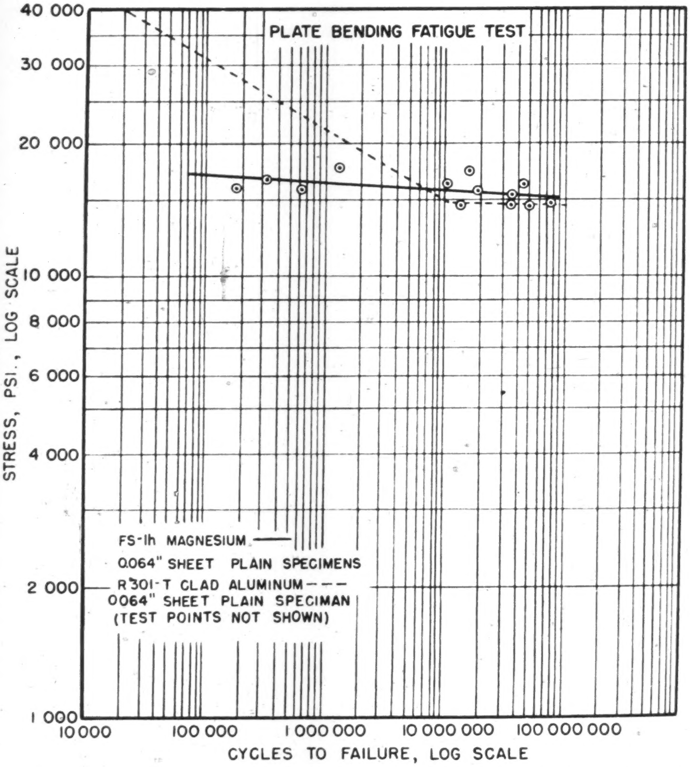

FIG. 8.—Fatigue Curves from Plate Bending Fatigue Tests on FS-1h and R301-T Sheet Specimens.

The notch sensitivity of wrought magnesium and aluminum alloys in general is greater than for the cast alloys, as is evidenced by the lower notch factor values. The notch factor values were between approximately 0.50 and 0.65 for both aluminum- and magnesium-base alloys at long life and in the short-life range the notch factors were slightly higher, between 0.53 and 0.71.

Plate Bending Tests:

The flat specimens tested in plate bending (flexural) fatigue were milled as shown in Fig. 2. The milled edges were abraded with metallographic abrasive paper to eliminate the machining marks. The flat surfaces of sheet and extrusions were untouched.

The data reported for these tests are for completely reversed stressing, that is, with a mean stress of zero. Results on several alloys are presented in Table V, results for sheets of thin gage and of a heavier gage being given for both magnesium and aluminum. Likewise, results on a series of extruded magnesium flats and on cast panels of C-HTA and 195-T6 alloys which had machined and polished surfaces are also summarized. All of the data presented on sheet were obtained by stressing in a direction longitudinal to the rolling direction. The results of tests on plain and notched specimens stressed transversely to the rolling direction were practically identical to those presented. The possible experimental error was estimated to be about ±1000 psi. for this test. Typical curves are shown in Fig. 8.

A tendency among the magnesium sheet alloys was noted for the annealed tempers to be slightly weaker than the hard tempers for both short and extended life ranges. With the exception of Ma and Mh sheet, the long-life properties of plain and notched bars were about the same for J-1, FS-1, and JS-1 alloys, Ma and Mh being considerably weaker. In short-life properties they are only slightly inferior. The thick and thin gages of the strongest aluminum sheet alloys were about a third stronger at short life than the corresponding magnesium sheet alloys. At 10^8 cycles, the plain-bar fatigue properties of the best alloys were about the same.

TABLE VI.—ROTATING-BEAM AND PLATE BENDING FATIGUE TEST RESULTS FOR THE SAME MATERIAL.

Alloy	Gage	Test	Plain Bar Properties		
			10^5	10^7	10^8
J-1h	0.500	Rotating Beam	25 000	19 000	19 000
J-1h	0.250	Plate Bend	19 000	14 000	14 000

It may be noted that the plain-bar data for thin gage specimens were generally higher than those for the thicker gages, this being particularly noticeable in the short-life range. Since there is no correlatable variation in other properties or metallurgy of the test bar material, and since inertia effects had been carefully minimized for tests on specimens of both gages, this is believed to be a manifestation of a mechanical size effect on the testing.

Tests to illustrate the effect of edge-notches were run on the thin gages only (S.C.F. = 1.6).[5] Alloys FS-1a, FS-1h, and 24S-T comprised a group least sensitive to such a notch at short life, FS-1a, J-1a and 24S-T being least sensitive for long-life considerations. M alloy sheet was quite sensitive to this type of notch.

The decrease in dynamic strength due to surface notches simulating surface scratches was slight for the magnesium sheet alloys. Indeed, in several instances no decrease in strength based on the unnotched cross-section was observed as a result of the notching. Although the aluminum sheet alloys were generally less sensitive to the surface notches than the magnesium sheet, the bending fatigue properties of neither aluminum nor magnesium sheet were affected greatly by surface scratches.

In tests on extruded flats prepared for testing according to the same schedule used for the sheet specimens, results showed that these alloys have about the same properties as in the corresponding annealed sheet condition, the effect of the increased section thickness being taken into account.

In tests conducted on cast magnesium C-HT and aluminum 195-T6 alloy panels, the specimens were milled to the same contour as the 0.064-in. sheet specimens. The surfaces were then machined smooth and polished to a specular finish as on the rotating-beam specimens. The results (Table V) showed that at short life 195-T6 was about a third stronger than C-HT. At 10^7

cycles the fatigue strength of the C-HT was about 20 per cent higher than the aluminum.

Since bending fatigue data as well as rotating-beam fatigue data are presented for two casting and five extrusion alloys, it is possible to compare the effects of the two different types of loading on the results. It will be observed that the 195-T6 aluminum alloy has very similar plate bending and rotating-beam fatigue properties. The 24S-T rotating-beam data are equal to the plate bending data for 0.020-in. gage 24S-T sheet, but are 4000 to 12,500 psi. higher than for 0.064-in. gage. The C-HT has plate-bending fatigue results that are 5000 to 6000 psi. lower than the rotating-beam results. Similarly, the plate-bending fatigue results of the extruded alloys are lower than the rotating-beam results. Except for the case of FS-1 which is 9000 to 10,000 psi. lower in plate-bending than in rotating-beam fatigue tests, the higher strength extrusion alloys, J-1 and O-1, are about 7000 psi. lower in plate bending than in rotating-beam fatigue throughout the entire life range, irrespective of stress level. Proof that these differences are, at least in part, not due to metallurgical differences between the flats and the rounds is given in Table VI. J-1h plate was tested under each of the two types of fatigue loading. The rotating-beam bars were cut from the 0.500-in. plate. The results show a 5000 to 6000 psi. difference throughout the life range.

TABLE VII.—AXIAL LOADING FATIGUE DATA FOR SHEET ALLOYS AT ROOM TEMPERATURE.[a]

Alloy and Temper	Plain Bar Properties $R = 0.25$[b]			Plain Bar Properties $R = 0.50$			Notched Bar Properties Symmetrical Surface Notch $R = 0.25$			Notch Factors Symmetrical Surface Notch $R = 0.25$		
	10^5	10^6	10^7	10^5	10^6	10^7	10^5	10^6	10^7	10^5	10^6	10^7
J-1a	24 000	20 000	18 000	31 000	24 000	19 000	18 500	17 000	16 500	0.77	0.85	0.92
J-1h	24 000	21 000	21 000	34 000	25 000	23 000	24 000	21 000	20 500	1.00	1.00	0.98
FS-1a	21 500	21 000	21 000	28 000	27 000	26 000	21 000	18 000	17 000	0.93	0.86	0.81
FS-1h	24 000	23 000	23 000	28 000	26 000	26 000	23 500	18 000	17 500	0.98	0.78	0.76
Ma	17 000	14 500	14 500	27 000	22 000	22 000	14 000	13 500	13 000	0.82	0.93	0.90
Mh	27 000	23 500	23 000	32 000	27 000	26 000	22 500	19 000	17 500	0.83	0.81	0.76
JS-1h	34 000	29 000	28 000	.....	.....	.....	31 000	25 500	24 000	0.91	0.88	0.86
75S-T clad	50 000	31 000	28 000	62 000	48 000	40 000	.....	.....	.....	...	...	...
R301-T clad	55 000	38 000	35 000	64 000	51 000	42 000	.....	.....	.....	...	...	...
24S-T	58 000	40 000	27 000	62 000	50 000	41 000	45 000	37 500	34 000	0.78	0.94	1.26

[a] Sheet gage for all alloys is 0.064 in.

[b] R = Ratio of minimum stress to maximum stress in each loading cycle.

TABLE VIII.—AXIAL LOADING FATIGUE DATA FOR SHEET ALLOYS WITH SPECIAL NOTCHES AT ROOM TEMPERATURE.[a]

Alloy and Temper	Notched Bar Properties Center Hole Notch $R = 0.25$			Notch Factors Center Hole Notch $R = 0.25$			Notched Bar Properties Eccentric Surface Notch $R = 0.25$			Notch Factors Eccentric Surface Notch $R = 0.25$		
	10^5	10^6	10^7	10^5	10^6	10^7	10^5	10^6	10^7	10^5	10^6	10^7
J-1h	20 500	18 000	17 500	0.85	0.86	0.83	.....	.....	.....	...	...	...
FS-1h	20 000	15 500	15 500	0.83	0.67	0.67	20 000	17 500	17 500	0.83	0.76	0.76
Mh	20 000	15 500	13 000	0.74	0.66	0.57	.....	.....	.....	...	...	...
24S-T	32 000	23 500	23 000	0.55	0.57	0.85	46 000	36 000	34 000	0.79	0.88	1.26

[a] Sheet gage for all alloys is 0.064 in.

Axial Loading Fatigue Tests:

Both sheet and rounds were tested in axial loading. The sheet specimens were prepared according to the same procedure used for the plate bending specimens and the rounds were turned and polished similarly to the rotating-beam test bars (Fig. 3). Test data are given in Tables VII, VIII, IX, and X.

Since axial-loading tests on sheet at a mean stress of zero would involve bending and, hence, nonaxial loading unless specially developed fixtures are used, the data presented are for tension stresses only. Two combinations of stress levels were used. These are expressed by an *R* value which denotes the ratio of the minimum to the maximum dynamic stress for any complete cycle of stressing: for sheet, *R* values of 0.25 and 0.50 and for rounds, *R* values of 0 and 0.25.

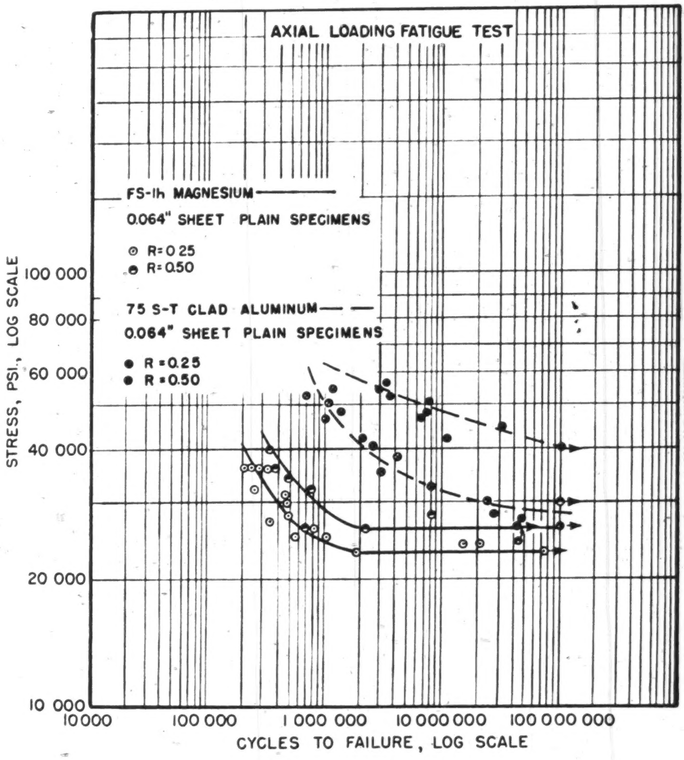

FIG. 9.—Fatigue Curves from Axial Loading Fatigue Tests on FS-1h and 75S-T Clad Sheet Specimens.

It should be noted that the typical fatigue curves for axial-loading tests on magnesium sheet, Fig. 9, have steeper slopes in the low cycle range than do the curves for bending tests. They also flatten out into horizontal run-out regions at about 10^6 cycles. Similar curves for aluminum sheet have no knee or horizontal portion below 10^7 cycles.

The estimated possible experimental error for these tests was ±2000 psi. For extended life, at either *R* values, the aluminum and the strongest magnesium sheet alloys, J-1h, FS-1h, FS-1a, Mh, and JS-1h, approached equality in fatigue strength (see Table VII). At short life, however, the magnesium sheet alloys of highest strength were 50 to 60 per cent as strong in fatigue as 24S-T sheet. In comparison to this difference it may be noted that all magnesium sheet alloys, except Ma which was lower and JS-1h which was higher, had about the same plain-bar fatigue properties in short life. Whether the specimens were longitudinal or transverse with respect to the rolling direction had no significant effect.

Data are given in Table VII on surface notched specimens run at an *R* value of 0.25. At short life, 24S-T appeared appreciably less sensitive to the notch than the least sensitive magnesium sheet alloys. The decrease in strength resulting from this notching was greater in the axial loading than in the bending tests.

Data on the sensitivities to a center hole notch of three "h" temper magnesium and 24S-T sheet alloys are given in Table VIII. (S.C.F. approximately 2.6).[5] Again J-1h appeared less notch sensitive than FS-1h and Mh at long life, and aluminum was considerably affected at short life.

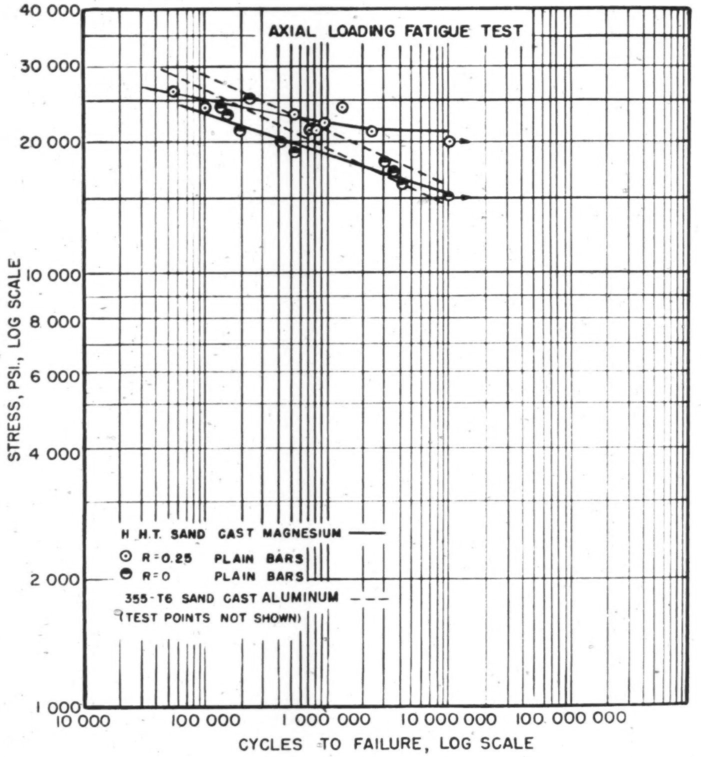

FIG. 10.—Fatigue Curves from Axial Loading Fatigue Tests on H-HT and 355-T6 Cast Test Specimens.

Specimens in FS-1h and 24S-T alloys were prepared with eccentric surface notches, that is, a notch scribed into one surface only of each specimen. At short life both alloys were equally sensitive to this notch; at longer life 24S-T was insensitive to this notch.

Axial-fatigue tests were conducted on cast rounds of magnesium and cast H alloy in each of its commercial heat treatments and on aluminum 195-T6

TABLE IX.—AXIAL LOADING FATIGUE DATA FOR CAST AND WROUGHT ROUNDS.

Alloy and Heat Treatment	Plain Bar Properties $R = 0.25$ Room Temperature			Notched Bar Properties $R = 0.25$ Room Temperature			Notch Factors $R = 0.25$ Room Temperature			Plain Bar Properties $R = 0$ Room Temperature			Notched Bar Properties $R = 0$ Room Temperature			Notch Factors $R = 0$ Room Temperature		
	10^5	10^6	10^7	10^5	10^6	10^7	10^5	10^6	10^7	10^5	10^6	10^7	10^5	10^6	10^7	10^5	10^6	10^7
ROOM TEMPERATURE CASTING ALLOYS																		
H-AC	24 000	21 500	19 000	16 500	14 000	12 000	0.69	0.66	0.63	22 000	20 000	18 000	16 000	13 000	11 000	0.73	0.65	0.61
H-ACS	26 000	23 000	21 000	18 500	16 000	14 000	0.71	0.70	0.66	23 000	21 000	19 000	16 500	13 500	11 000	0.72	0.64	0.58
H-HT	25 000	22 500	21 000	20 500	15 000	15 000	0.82	0.67	0.71	23 000	19 000	15 000	18 000	15 500	14 000	0.78	0.82	0.93
H-HTS	29 000	26 000	24 000	20 000	18 000	18 000	0.69	0.69	0.75	24 000	21 000	21 000	18 000	15 500	13 500	0.75	0.74	0.64
H-HTA	25 000	23 000	21 000	21 000	18 000	18 000	0.84	0.78	0.86	28 000	21 000	20 000	17 000	14 000	11 500	0.61	0.67	0.58
355-T6	28 000	21 000	15 500	26 500	20 000	15 000	0.95	0.95	0.97	26 000	19 000	14 000	23 000	15 000	8 000	0.89	0.79	0.57
195-T6	34 000	26 000	20 000	27 000	20 000	15 000	0.79	0.77	0.75	33 000	24 500	18 000	23 500	17 000	12 000	0.71	0.69	0.67
WROUGHT ALLOYS																		
O-1HTA	39 000	36 500	34 000	21 000	20 000	19 000	0.54	0.55	0.56	39 000	35 500	32 000	18 500	17 000	16 000	0.47	0.48	0.50
24S-T	58 000	52 000	46 000	38 000	26 000	26 000	0.66	0.50	0.56	56 000	47 000	40 000	31 000	25 000	25 000	0.55	0.53	0.63
ELEVATED TEMPERATURE, 275 F. CASTING ALLOYS																		
H-HTS	24 000	22 000	20 000	19 000	15 000	12 000	0.79	0.68	0.60	21 500	20 000	18 500	16 000	11 000	10 000	0.75	0.55	0.54
H-HTA	25 000	24 000	23 000	18 000	16 500	15 000	0.72	0.69	0.65	22 000	20 000	18 000	15 500	14 000	12 500	0.70	0.70	0.69
355-T6	25 000	19 000	15 000	23 000	18 000	14 000	0.92	0.95	0.93	24 000	15 500	10 000	22 000	15 500	11 000	0.92	1.00	1.10

and 355-T6 at R values of 0 and 0.25. The test results at room temperature, (Table IX and Fig. 10) bear about the same relation to each other as in the rotating-beam tests.

The magnesium alloys were sensitive to notches in these tests to about the same degree as in the rotating-beam tests. Although 195-T6 alloy was less notch sensitive than magnesium at $R = 0.25$, in general the aluminum seemed to be slightly more notch sensitive in these tests than in the rotating-beam tests.

Axial fatigue tests run at 275 F., Table IX, revealed the same relative results between magnesium and aluminum alloys as in axial and rotating-beam tests at room temperature. This comparison, however, was not as much in favor of aluminum in the axial loading tests as it is in the rotating-beam tests at 275 F.

TABLE X.—AXIAL LOADING FATIGUE TESTS ON RIVETED JOINTS.

Alloy	Rivet Disposition	Sheet Stress, psi. ($R = 0.25$)		
		10^5	10^6	10^7
FS-1h	6 in single row	8 500	5000	4500
FS-1h	12 in double row	9 000	7000	7000
24S-T........	6 in single row	13 500	8000	4500
24S-T........	12 in double row	15 000	9500	6000

Axial fatigue tests at room temperature on extruded rod specimens showed that the 24S-T maintained superiority in plain-bar and notched-bar strength over O-1HTA throughout the life range tested. The notch sensitivities of the two alloys were comparable.

As stated in the introduction, the fatigue properties of joints connecting wrought materials, rather than the fatigue properties of unnotched specimens, are more significant for indicating the serviceability characteristics of the metal. Accordingly, data from axial-loading tests on two simple types of riveted joints are presented in Table X. Single and double rivet row joints of 24S-T and FS-1h were made according to recommended joint design practice with $\frac{15}{32}$-in. rivet spacing in the 3.16 by 12-in. specimens of 0.40-in. sheet with 17S-T rivets $\frac{5}{32}$ in. in diameter.

It will be noted that the fatigue properties of the joints were, in some cases, less than 25 per cent of the strength of the sheet of which they were made. It will also be observed that the fatigue strength of the magnesium joints at long life was equal to or superior to that of the aluminum joints. For the short-life range, the aluminum joints were stronger and were 31 per cent of the monoblock strength. It is evident that prediction of this behavior could not be made from the data for the monoblock and the notched specimens.

DISCUSSION

The service experiences of most complex metal parts are not readily amenable to a simplified analytical segregation of variables or to the assessment of these variables in terms of their relative importance. It is for this reason that test bar fatigue data, which represent an attempt to evaluate the reaction of various metals to segregated variables, may be of little use for predicting serviceability unless very cautiously appraised. The value of the test bar data for evaluating alloys for serviceability thus seems to be proportional to the number and diversity of the various conditions under which the testing was performed. The most successful utilization of the test bar data presented for comparing alloys and determining serviceability characteristics has been to survey comprehensively the data in their entirety, that is, as affected by the mode of load application, degree and kind of stress concentration, life range, temperature and mean stress level, and to arrive at general conclusions only. When this practice is followed, such important variables as the mode of load application, the degree of stress concentration (the effects of both of which have been demonstrated in Table VI and in Fig. 6), and the effects of various surface finishes are lumped together and diluted, just as they are in service, to permit generalizations. Qualitatively—that is, for alloy comparisons and for general design recommendations—this treatment of the data has been found to lead to conclusions that have been verified in recent service experience. Quantitatively—that is, for specific design values—however, this and other uses of fatigue data from test bars have been found by experience to be of very limited value. Fatigue tests run on actual parts during which experimental stress measurements have been made have given the most useful quantitative information for service application.

In conformity with the above recommendations for the use of the test bar fatigue data for the light metals, general conclusions are forthcoming from the test bar data presented. First, however, a review of the test bar results is necessary.

For cast alloys in room-temperature tests in the short-life range and under conditions of no stress concentration, it is apparent that the strongest aluminum alloys have from 0 to 30 per cent greater dynamic strength than the strongest magnesium alloys. The exact comparison depends upon the type of dynamic loading test and the specific alloys selected for the comparison. At extended life, the comparison is reversed and magnesium is from 5 to more than 50 per cent stronger than aluminum.

At 275 F. under conditions of no stress concentration, aluminum is from 0 to 30 per cent stronger at short life. The strongest magnesium alloys are from 0 to 80 per cent stronger in fatigue at long life. The comparison appears dependent on either the type of test considered or the mean stress (static load or pre-stress which is superimposed on the dynamic load). The data for elevated temperature from the axial-loading tests, which are for higher mean stress than are the rotating-beam data, show the magnesium casting alloys to have greater superiority at long life over the aluminum tested than is evident in the rotating-beam data.

Since most parts break at points of stress concentration, the notched-bar values are of particular significance. For short life at room temperature, the notched-bar strengths (S.C.F. = 2) of the strongest aluminum alloys are up to 30 per cent higher than are those of the strongest, notched, magnesium casting alloys. For long life at room temperature, the notched-bar strength is about the same for both aluminum- and magnesium-base casting alloys when the mean stress is near zero, as it is in rotating-beam tests. The magnesium-base casting alloys, however, are considerably stronger than aluminum at long life in dynamic notch strength in the axial loading tests. For notches of S.C.F. greater than 2, cast aluminum appears much superior to cast magnesium.

At 275 F., the aluminum alloys are from 25 to 40 per cent higher in notched-bar strength at short life and from 0 to about 40 per cent higher at long life. Here again, the data from the axial loading tests define the lower limits of this comparison, while the rotating-beam data give the upper limits or show the greatest superiority of aluminum.

The general conclusions regarding serviceability to be derived from the above observations on test bars are as follows:

Because of the high notch sensitivity of the magnesium-base casting alloys to severe stress concentrations, regions of excessively high stress concentration should be avoided in magnesium castings. Stress concentrations that involve an S.C.F. greater than 2, however, do not often exist in castings. When a severe stress concentration does exist, such as around bolt holes and threads, the nominal stress in the region should be maintained low so as to render the stress concentration relatively unimportant. For elevated temperature applications, the avoidance of serious stress raisers is of particular importance. These facts have been recognized by the designers and users of magnesium castings and have been dealt with successfully by observing recommended design practices. These recommendations pertain to such subjects as the use of generous fillets and bosses and also of gradual contour changes, as well as care in threading and terminating stud holes. Surface finishes reasonably free from local irregularities are also to be recommended for extended life.

It may be concluded, finally, from the data from cast test bars that, if stress concentrations are kept at a reasonable minimum, in cases where the full 33 per cent weight saving of cast magnesium over cast aluminum cannot be realized on an equal strength basis, at least an appreciable weight saving can be gained.

A review of the fatigue data presented for the wrought metal alloys shows that, in general, equally as great sensitivity to notches in fatigue is manifest in the wrought state for both aluminum and magnesium. This high notch sensitivity of these wrought metal alloys in comparison to the cast alloys is counteracted in part, however, by the higher, absolute, test bar fatigue properties of the wrought metal. These two characteristics compensate each other in the rotating-beam tests to the extent that in most cases the notched bar strengths for the wrought metals at an S.C.F. = 2 are approximately equal to the notched-bar strengths of the cast alloys for the same stress concentration. In the axial loading fatigue tests, the wrought alloys appear considerably stronger than the cast alloys.

Irrespective of the type of dynamic loading test, wrought aluminum has greatest test bar strength superiority at the short-life end of the fatigue curves. This is most clearly evident in the axial loading tests where the aluminum unnotched sheet specimens are about 60 per cent stronger than the strongest magnesium sheet at 10^5 cycles. At extended life, however, the wrought magnesium and aluminum alloys have dynamic test bar properties that approach equality. That this appears to be the case also for fabricated assemblies containing wrought alloys is demonstrated in the fatigue data obtained for joints of standard design.

CONCLUSIONS

1. In general, the fatigue curves of aluminum are steeper than are those of magnesium and also do not show to the same degree the tendency to flatten out in the lower stress ranges as do the curves for magnesium alloys.

2. In consequence of the preceding conclusion, accelerated tests at high overloads may favor the cast and wrought aluminum alloys over the equivalent magnesium alloys. For this reason the results of accelerated tests comparing magnesium and aluminum alloys must be considered with caution.

3. At lower stresses, cast magnesium alloys have notched bar strengths that are comparable to those of cast aluminum alloys and unnotched bar strengths that are superior.

4. At the lower stresses, the wrought

magnesium and wrought aluminum alloys have comparable notched and plain bar strengths, except for rounds in the axial loading tests, which showed higher plain and notched bar strengths for wrought aluminum.

5. Except for cases of extreme stress concentrations at notches, wrought magnesium and aluminum alloys have comparable notch sensitivities in fatigue loading. For severe stress concentrations (above S.C.F. of 2) wrought aluminum is less notch sensitive than wrought magnesium.

6. At moderate stress concentrations cast magnesium alloys tend to be slightly more notch sensitive than cast aluminum alloys. Under severe concentrations of stress (S.C.F. greater than 2), cast aluminum is markedly less notch sensitive.

7. The fatigue properties of the common commercial magnesium alloys appear somewhat more sensitive to increases in temperature than are those of the aluminum alloys. This effect was least evident in the axial loading tests.

8. Experience shows that quantitative use for design purposes of the test bar values cannot be made. However, it is evident from the data that efforts should be made to minimize stress concentrations in the design of any parts, particularly magnesium alloy parts. If this is done, test bar data indicate that, in particular cases where the full 33 per cent weight advantage of magnesium over aluminum alloys cannot be realized on the basis of equality of fatigue properties, at least an appreciable weight saving is possible with equivalent serviceabilities. In general, the cases where the full weight advantage of magnesium is unattainable are where high stress concentrations, short-life applications, or elevated temperatures are involved.

DISCUSSION

CHAIRMAN H. F. MOORE.[1]—Were those theoretical stresses or actual?

MR. GEORGE H. FOUND (*author*).—The former, theoretical.

I should like to remark, with regard to a criticism made of the paper by Messrs. Jackson and Grover,[2] that I believe aircraft designers—at least those who are beginning to manufacture commercial planes, rather than war planes—are very definitely interested in long-life properties, as well as short-life properties.

MR. LEO SCHAPIRO.[3]—The author's *S-N* diagram in Fig. 5 shows a flattening at about ten million cycles, and he makes the statement that the flattening is characteristic. Judging by the markings "HT," I presume the materials were heat treated. The paper by Messrs. Jackson and Grover,[2] dealing with magnesium alloys in sheet form, showed no flattening of either the heat-treated or nonheat-treated materials. Does the flattening characterize magnesium alloys in general, or just magnesium castings?

MR. FOUND.—The correct statement would be that the fatigue curves of magnesium alloys show a tendency to flatten out. There are cases where they do not. There does not appear to be any correlation between this tendency and the heat treatment. The flattening of the *S-N* curves for magnesium is not limited to castings only. There was one case shown where the sheet alloy flattened out. (Note curve for FS-1h in Fig. 12.)

MR. JOHN M. LESSELLS.[4]—I should like to direct to the author the same question asked Mr. Jackson regarding the path of the fatigue crack in magnesium alloys—is it intercrystalline or transcrystalline?

It would be helpful also to have some further data as to how the stress concentration and strength-reduction factors were determined. Is it true that magnesium alloys appear to have greater strength-reduction factors than do aluminum alloys?

MR. SCHAPIRO.—You were first asking about the transcrystalline or intercrystalline crack path with regard to stress-corrosion cracking. You are now asking with regard to fatigue cracking. Please let us not mix the two together. The stress-corrosion failures which we have examined have always been intergranular.

With regard to aircraft structures, I believe I am safe in speaking for the aircraft manufacturers in saying that we are interested in cycles of life less than one million, and even less than one hundred thousand.

MR. RICHARD L. TEMPLIN.[5]—Practically all fatigure failures in aluminum structures are transgranular or transcrystalline and practically all failures in stress corrosion under conditions of static stress are intercrystalline. Under combined conditions of fatigue and stress corrosion, whether the failure is transcrystalline or intercrystalline depends on the predominant factor, that is, fatigue or corrosion. Either may be governing under some conditions, yet in others there might exist a combination of the two factors wherein failure could start primarily from corrosion as an intergranular fracture and subsequently, as the fatigue stresses become predominant, the fracture might change to a transcrystalline type. The important point is to avoid failure by either corrosion or fatigue as well as a combination of the two.

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[1] Research Professor of Engineering Materials, Emeritus, University of Illinois, Urbana, Ill.

[2] See p. 783.

[3] Douglas Aircraft Co., Inc., Santa Monica, Calif.

[4] Associate Professor of Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

[5] Assistant Director of Research and Chief Engineer of Tests, Aluminum Research Labs., Aluminum Company of America, New Kensington, Pa.

MR. FOUND.—Fatigue failures in magnesium at room temperature are always transcrystalline, as they are for aluminum. At elevated temperatures, in the vicinity of 300 F. and above, the failures tend to be intercrystalline. Stress-corrosion failures induced in magnesium under simulated atmospheric conditions also are always transcrystalline, which, in this case, is unlike aluminum. Static failures in magnesium are observed to be either trans- or intercrystalline, depending upon the structure of the magnesium as affected by heat treatment. The exact nature of any failure, that is, whether it is predominantly of a static, fatigue or stress-corrosion type, can be accurately deduced from the characteristics of the branch cracks emanating from the main failure. This subject is described in greater detail in a recent publication.[6]

With regard to determining the stress-concentration factors, we have done no theoretical work. We have used the literature and adopted test bar designs prescribed by those who have theoretically calculated and also experimentally determined the stress-concentration factors.

CHAIRMAN MOORE.—Did you follow the Neuber analysis?

MR. FOUND.—Yes, we have used that.

MR. WAYNE LEWIS.[7]—It is possible that too much emphasis is being placed on the endurance limit as the criterion of the suitability of a material or construction for aircraft. Possibly commercial aircraft manufacturers are not interested in the early portion of S-N curves, but the military air forces are.

It is reasonable to assume that different parts of an airplane will be subjected to a different number of repetitions of design stress during the normal or expected life of the airplane. For example: the wing structure will be loaded to the design stress a certain number of times during its life, while the landing gear may be loaded to design stress a greatern umber of times than the wing structure. The propellers would be loaded so many times to maximum that it would be necessary to keep the maximum actual stress below the endurance limit. To design an entire airplane on the basis of endurance limit would produce an unbalanced design and impose a weight penalty on the parts stressed to maximum the fewest number of times.

MR. FOUND (*author's closure*).—I should like to bring up this short and long-life problem of evaluation again. Neither should be emphasized at the expense of the other, and I do not believe I did that.

The long-life portion of the S-N curves is important. In a propeller, for instance, whether designed in magnesium or aluminum, it does not take long for many millions of cycles of alternating stress to be applied. This argument applies for other parts in the plane as well, particularly in the power plant.

Now let us consider the short life portion of the S-N curves. The number of cycles of alternating stress applied to many types of landing wheels is often only several million during their intended life. In these cases, it would be inefficient to carry extra weight in a design intended for longer life. On the other hand, certain landing wheels are used for many millions of cycles of alternating stress in which cases the operating firms may prefer to design for long life and pay for carrying the extra weight rather than to design for short life and change wheels periodically.

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[6] P. F. George, "Fracture Characteristics of Magnesium Castings," *Aluminum and Magnesium*, Vol. 1, No. 9, June, 1945, p. 14.

[7] Engineer, Forest Products Laboratory, Madison, Wis.

It is hoped that this discussion will point out that there are too many considerations in aircraft and other applications which may render critical either static loads or long life or short life alternating loads to justify a general evaluation of materials on the basis of only one of these factors alone.

THE FATIGUE PROPERTIES OF BERYLLIUM-COPPER STRIP AND THEIR RELATION TO OTHER PHYSICAL PROPERTIES*

By G. R. Gohn[1] and S. M. Arnold[1]

Synopsis

This paper describes the results of a series of fatigue tests made on several lots of beryllium-copper strip, ranging in thickness from 0.020 to 0.040 in. Specimens of several tempers were tested unaged and after a variety of precipitation-hardening treatments.

A marked improvement in fatigue strength of corresponding tempers tested in 1945 over those tested in 1936 was observed. Alloys containing as little as 1.80 per cent beryllium hardened satisfactorily and had essentially the same fatigue properties as those containing higher percentages of beryllium. Increasing the nickel content to 2 per cent did not impair the tensile or fatigue properties, provided the beryllium content was maintained at the maximum permitted by the specifications.

Material in the "as received" condition had essentially the same fatigue characteristics for a given temper, regardless of source or alloy composition. The same correlation was found to be true for the heat-treated material.

While overaging had little or no adverse effect on the fatigue properties, underaging, in most cases was deleterious. Both overaging and underaging had a marked effect on the other physical properties.

While buffing failed to improve the fatigue strength of heat-treated specimens, pickling, polishing, and short-time heat-treatments in salt and oil baths resulted in some improvement. The best fatigue results were obtained with specimens heat-treated and subsequently polished. High temperature, short-time heat-treatments developed better fatigue properties than did the more conventional heat-treatments without subsequent surface treatment.

In communication equipment it is necessary to use many switches, relays, jacks, and other pieces of electrical apparatus containing small, flat springs. One of the problems confronting the designer of this apparatus is the choice of a material suitable for the manufacture of such springs. The springs are generally blanked from thin metal sheet or strip and are frequently formed into rather intricate shapes in punch press or similar manufacturing operations. These parts often carry small electrical currents at relatively low voltages, contact being made either directly between two base metal springs or through precious metal contacts welded to them. The materials used, therefore, not only must combine high elastic and fatigue properties with good electrical conductivity and weldability, but also must be sufficiently ductile to permit forming without difficulty. Generally, the material must be nonmagnetic, corrosion-resistant and, in some cases, resistant to creep or drift under continuously applied loads, either at room or elevated temperatures. Low raw material and fabrication costs are also factors in the choice, since millions of these springs are used annually.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

[1] Members of Technical Staff, Bell Telephone Laboratories, Inc., New York, N. Y.

High-carbon steels are frequently unsuitable for use as springs in communication apparatus because they are magnetic and lack adequate corrosion resistance. In general, the three materials most widely used for these applications are the 5 and 8 per cent tin bronzes (commonly known as phosphor bronzes) and the 18 per cent nickel silver alloy. However, since the best combination of spring properties is obtained in these alloys only by cold-rolling, the forming characteristics are not too good in the tempers in which they are used. Therefore, a corrosion-resisting copper-base alloy which could be formed in the soft or annealed condition, and heat-treated after forming to obtain the desired spring characteristics, has long been sought. The comparatively recent development of the beryllium-copper alloys has made available a commercial alloy meeting many of the requirements prescribed.

REVIEW OF PREVIOUS WORK

No systematic study of beryllium or its alloys appears to have been made until 1916 when Oesterheld **(1)**[2] studied the structure of the beryllium-copper alloys and presented an equilibrium diagram which indicated that a precipitation-hardening alloy might be obtained by adding a small amount of beryllium to copper. In 1927, Illig **(2)** reported that beryllium-copper brush springs and contact elements used on electric locomotives had fatigue characteristics superior to those of phosphor bronze springs. No data on the fatigue strength of the beryllium-copper alloys are given in the literature until 1937, when Gough and Sopwith **(3)** reported that the fatigue strength of some lightly cold-worked and aged rod was 35,800 psi. at 55 million cycles. In the same year, Greenall and Gohn **(4)** reported that dispersion-hardened alloys of the beryllium-copper type were comparable in their minimum fatigue characteristics to cold-rolled, alloy C phosphor bronze (8 per cent tin) strip. Endurance limits at 10^8 cycles of reversed stress were found to vary from 23,000 to 36,000 psi. for the various lots of beryllium-copper strip studied.

In 1941, Anderson and Smith **(5)**, working with highly polished rod specimens, found that the inherent fatigue strength of the beryllium-copper alloys which they studied varied from 47,000 to 61,500 psi. at 3×10^8 cycles of reversed stress. To the best of our knowledge, no other test data on the fatigue strength of beryllium-copper alloys are available.

TESTING PROGRAM

To provide suitable data for the design of small flat springs, the Bell Telephone Laboratories, since 1927, have studied the fatigue characteristics of promising spring materials and attempted to correlate the fatigue strength with other physical properties. The studies included four separate investigations of the fatigue properties of beryllium-copper strip. These were:

1. The studies, previously reported by Greenall and Gohn **(4)**, made on binary and nickel-bearing ternary alloys aged at 525 F. for 1 and 2 hr.

2. Studies made in 1941 to evaluate the fatigue characteristics of a 1.85 per cent beryllium alloy as compared to the standard 2.00 per cent alloy. These tests were undertaken at the time A.S.T.M. Committee B-5 on Copper and Copper Alloys was considering a reduction in the minimum beryllium content from 1.90 to 1.85 per cent. The tests

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 775.

TABLE I.—CHEMICAL ANALYSES.

Designation	Beryllium, per cent	Nickel, per cent	Cobalt, per cent	Iron, per cent	Additive Elements, Nickel + Cobalt + Iron, per cent	Copper, per cent	Copper + Additive Elements + Beryllium, per cent	Aluminum, per cent	Silicon, per cent	Remarks
1936 INVESTIGATION										
B1-36	2.25	....	....	....	....	Remainder	....	....	....	Nominal composition
B2-36	2.15	0.31	....	0.06	0.37	97.44	99.96	....	0.03	
B3-36	2.16	0.32	....	0.06	0.38	97.50^a	100.04^a	....	0.03	
B4-36	2.14	0.28	....	0.06	0.34	97.54^a	100.02^a	....	0.03	
A1-36	2.25	0.50	....	....	....	Remainder	....	....	....	Nominal composition
A2-36	2.25	0.50	....	....	....	Remainder	....	....	....	Nominal composition
A3-36	2.25	0.50	....	....	....	Remainder	....	..	....	Nominal composition
1941 INVESTIGATION										
C1-41	2.00	....	0.25	....	....	....	....	....	....	Nominal composition
C2-41	1.85	....	0.30	....	....	....	....	....	....	Nominal composition
1942 INVESTIGATION										
E1-42	2.25	....	....	....	....	Remainder	....	....	....	Nominal composition
D1-42	2.18	1.10	....	0.08	1.18	Remainder	....	....	....	
D2-42	1.94	1.17	....	0.10	1.28	Remainder	....	....	....	
D3-42	2.12	2.00	....	0.10	2.10	Remainder	....	....	....	
D4-42	1.98	2.13	....	0.11	2.24	Remainder	....	....	....	
1945 INVESTIGATION										
A4-45	2.12	0.28	<0.01	0.17	0.45	97.31	99.88	Nil	0.09	
A5-45	2.03	0.30	0.01	0.13	0.44	97.50^a	99.97^a	0.01	0.07	
A6-45	2.17	0.28	0.03	0.19	0.50	97.28^a	99.95^a	0.01	0.10	
C3-45	1.87	<0.01	0.10	0.12	0.22	97.91^a	100.00^a	Nil	0.11	
C4-45	1.88	<0.01	0.09	0.13	0.22	97.88^a	99.88^a	Nil	0.11	
C5-45	1.86	<0.01	0.08	0.12	0.20	97.89^a	99.95^a	Nil	0.11	
E2-45	1.90	0.49	0.04	0.16	0.69	97.26	99.85	0.02	0.11	
E3-45	1.81	0.49	0.04	0.15	0.68	97.31	99.80	0.04	0.12	
E4-45	1.89	0.40	0.03	0.12	0.55	97.44	99.88	Nil	0.05	
E5-45C	1.88	0.27	0.07	0.07	0.41	97.19	99.48	0.24	0.26	
E6-45C	1.87	0.27	0.07	0.07	0.41	97.17	99.45	0.22	0.26	
E7-45C	1.88	0.28	0.07	0.07	0.42	97.16	99.46	0.22	0.26	
Specification Requirements										**Impurities, per cent**
A.S.T.M. B 120 – 41 T (Replaced by B 194 – 45 T)	1.90 to 2.20	....	....	....	0.50 max.	Remainder	....	....	....	0.50 max.
A.S.T.M. B 194 – 45 T	1.90 to 2.15	....	....	....	0.15 to 0.50	....	99.50 min.	....	....	....

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[a] Copper appears to be high since total of determined elements exceeds 100 per cent. However, error is less than precision of analytical method employed.

TABLE II.—PHYSICAL PROPERTIES.

Designation	Nominal Composition, per cent	Con-dition	Precipitation-Hardening Treatment	Nominal Thick-ness, in.	Direction of Rolling with Respect to Length of Specimen	Tensile Strength, psi.	Propor-tional Limit, psi.	Yield Strength at 0.01 per cent Offset, psi.	Modulus of Elasticity, psi.	Elonga-tion in 2 in., per cent	Rockwell Hardness	Fatigue Strength at 10^8 Cycles of Reversed Stress, psi.	Ratio of Fatigue Strength to Tensile Strength
1936 INVESTIGATION													
B1-36-V[a]	2.25 Be	AT	2 hr. at 525 F.	0.032	Parallel	171 200	....	....	19 100 000	4.8	B110.9	31 000	0.18
B2-36-V[a]	2.25 Be, 0.30 Ni	AT	2 hr. at 525 F.	0.032	Parallel	....	....	....	....	....	....	28 000	....
B3-36-V[a]	2.25 Be, 0.30 Ni	½HT	2 hr. at 525 F.	0.032	Parallel	173 000	....	....	....	....	....	27 250	0.16
B4-36-V[a]	2.25 Be, 0.30 Ni	HT	2 hr. at 525 F.	0.032	Parallel	....	....	....	....	....	....	28 000	....
A1-36	2.25 Be, 0.50 Ni	H	None	0.020	Parallel	....	....	....	....	....	....	27 000	....
A1-36-V	2.25 Be, 0.50 Ni	HT	2 hr. at 525 F.	0.020	Parallel	....	....	....	....	....	....	36 000	....
A2-36	2.25 Be, 0.50 Ni	½H	None	0.032	Parallel	....	....	....	....	....	....	23 250	....
A2-36-V	2.25 Be, 0.50 Ni	½HT	2 hr. at 525 F.	0.032	Parallel	....	....	....	....	....	....	25 500	....
A3-36	2.25 Be, 0.50 Ni	½H	None	0.020	Parallel	84 000	25 900	....	19 400 000	19.1	B85.9	23 000	0.27
A3-36-V	2.25 Be, 0.50 Ni	½HT	2 hr. at 525 F.	0.020	Parallel	171 000	49 500	....	19 800 000	4.0	B107.6	26 500	0.16
A3-36-VI	2.25 Be, 0.50 Ni	½HT	1 hr. at 525 F.	0.020	Parallel	....	....	....	....	....	....	30 000	....
1941 INVESTIGATION													
C1-41-VII	2.00 Be, 0.25 Co	AT	3 hr. at 600 F. in salt bath	0.032	Parallel	184 000	68 000	82 500[b]	19 500 000	3.0	C41	26 000	0.14
C1-41-VIII	2.00 Be, 0.25 Co	½HT	2 hr. at 600 F. in salt bath	0.032	Parallel	199 000	70 000	90 000[b]	20 500 000	2.0	C42	34 700	0.17
C1-41-VIII	2.00 Be, 0.25 Co	HT	2 hr. at 600 F. in salt bath	0.032	Parallel	204 500	74 000	92 000[b]	20 000 000	2.0	C43	39 000	0.19
C2-41-VII	1.85 Be, 0.3 Co	AT	3 hr. at 600 F. in salt bath	0.032	Parallel	169 600	42 000	52 000[b]	19 500 000	5.0	C37	25 000	0.15
C2-41-VIII	1.85 Be, 0.3 Co	½HT	2 hr. at 600 F. in salt bath	0.032	Parallel	189 100	45 900	59 000[b]	20 000 000	4.0	C41	38 000	0.20
C2-41-VIII	1.85 Be, 0.3 Co	HT	2 hr. at 600 F. in salt bath	0.032	Parallel	192 900	55 000	62 000[b]	19 700 000	2.5	C41.5	38 500	0.20
1942 INVESTIGATION													
E1-42-IX	2.25 Be	½HT	1 hr. at 600 F. in salt bath	0.040	Parallel	189 400	77 000	88 600	19 200 000	4.0	C41	43 500	0.23
D1-42-IX[a]	2.20 Be, 1.0 Ni	½HT	1 hr. at 600 F. in salt bath	0.040	Parallel	183 100	87 900	105 800	19 700 000	4.7	C41	44 500	0.24
D2-42-IX[a]	2.00 Be, 1.0 Ni	½HT	1 hr. at 600 F. in salt bath	0.040	Parallel	166 300	80 100	98 200	19 200 000	11.2	C37	39 200	0.23
D3-42-IX[a]	2.20 Be, 2.00 Ni	½HT	1 hr. at 600 F. in salt bath	0.040	Parallel	162 800	82 600	105 400	18 400 000	8.5	C37	35 700	0.22
D4-42-IX[a]	2.00 Be, 2.00 Ni	½HT	1 hr. at 600 F. in salt bath	0.040	Parallel	154 300	76 700	94 500	19 500 000	13.7	C36	43 000	0.28

A4-45	2.00 Be, 0.35 Ni	A	None	0.032	Parallel	76 500	19 800	28 300	16 100 000	39.3	B68.2	32 000	0.42
					45 deg.	74 500	19 100	26 900	17 800 000	43.8	B68.2		....
C3-45	2.00 Be, 0.15 Co	A	None	0.032	Parallel	67 800	14 900	19 300	16 700 000	56.2	B55.2	32 500	0.48
					45 deg.	66 400	14 500	19 800	16 800 000	55.5	B55.2	....	....
E2-45	2.00 Be, 0.35 Ni	A	None	0.032	Parallel	72 300	15 900	23 200	17 300 000	42.4	B60.9	33 000	0.46
					45 deg.	70 100	20 900	26 200	16 000 000	44.1	B60.9	....	....
E5-45 C	2.00 Be, 0.35 Ni	A	None	0.032	Parallel	72 000	20 500	27 300	18 600 000	55.1	B62.3	32 500	0.45
A4-45-I	2.00 Be, 0.35 Ni	AT	3 hr. at 600 F. in purified nitrogen	0.032	Parallel	181 300	73 100	90 700	19 100 000	7.8	C37.2	35 500	0.2
C3-45-I	2.00 Be, 0.15 Co	AT	3 hr. at 600 F. in purified nitrogen	0.032	Parallel	154 200	45 600	60 700	19 800 000	7.9	C31.7	34 800	0.23
E2-45-I	2.00 Be, 0.35 Ni	AT	3 hr. at 600 F. in purified nitrogen	0.032	Parallel	151 300	50 200	69 900	19 500 000	6.6	C30.5	34 500	0.23
E5-45 C-XVI	2.00 Be, 0.35 Ni	AT	3 hr. at 600 F. in muffle furnace	0.032	Parallel	177 000	56 300	87 600	19 600 000	6.8	C37.7	36 000	0.20
C3-45-III	2.00 Be, 0.15 Co	AT	3 hr. at 575 F. in purified nitrogen	0.032	Parallel	173 400	70 900	89 900	18 900 000	7.6	C35.5	31 700	0.18
A4-45-XII	2.00 Be, 0.35 Ni	AT	3 hr. at 600 F. in muffle furnace	0.032	Parallel	170 500	73 500	86 800	18 000 000	5.6	30N60.6	43 000^c	0.25
					45 deg.	164 100	83 600	91 100	16 800 000	6.8	30N60.6	....	....
C3-45-XII	2.00 Be, 0.15 Co	AT	3 hr. at 600 F. in muffle furnace	0.032	Parallel	132 400	63 600	69 500	17 600 000	10.8	30N50.6	47 000^c	0.36
					45 deg.	128 300	54 700	62 600	18 900 000	7.0	30N50.6	....	....
E2-45-XII	2.00 Be, 0.35 Ni	AT	3 hr. at 600 F. in muffle furnace	0.032	Parallel	144 300	65 000	73 200	17 200 000	7.5	30N54.6	40 400^c	0.28
					45 deg.	142 100	60 600	70 200	18 000 000	7.0	30N54.6	....	....
A5-45	2.00 Be, 0.35 Ni	½H	None	0.032	Parallel	109 000	59 600	72 600	17 600 000	5.5	B98.0	34 800	0.32
					45 deg.	107 500	58 000	68 000	16 200 000	7.2	B98.0	....	....
C4-45	2.00 Be, 0.15 Co	½H	None	0.032	Parallel	85 100	48 400	58 900	17 400 000	16.0	B88.0	34 000	0.40
					45 deg.	83 200	48 800	56 500	16 600 000	18.0	B88.0	....	....
E3-45	2.00 Be, 0.35 Ni	½H	None	0.032	Parallel	89 500	45 400	60 800	18 700 000	16.4	B90.1	35 000	0.39
					45 deg.	84 700	44 600	54 800	16 800 000	18.1	B90.1	....	....
E6-45 C	2.00 Be, 0.35 Ni	½H	None	0.032	Parallel	98 000	28 500	48 600	18 900 000	16.0	B80.0	38 500	0.39
A5-45-II	2.00 Be, 0.35 Ni	½HT	2 hr. at 600 F. in purified nitrogen	0.032	Parallel	200 100	80 100	113 500	18 600 000	3.0	C41.5	38 500	0.19
C4-45-II	2.00 Be, 0.15 Co	½HT	2 hr. at 600 F. in purified nitrogen	0.032	Parallel	174 300	49 900	82 700	19 500 000	3.8	C36.7	39 000	0.22
E3-45-II	2.00 Be, 0.35 Ni	½HT	2 hr. at 600 F. in purified nitrogen	0.032	Parallel	179 800	86 100	108 800	19 800 000	7.8	C37.7	35 000	0.19
E6-45 C-XVII	2.00 Be, 0.35 Ni	½HT	2 hr. at 600 F. in muffle furnace	0.032	Parallel	176 500	67 400	85 400	19 800 000	5.6	C37.7	44 000	0.25
C4-45-IV	2.00 Be, 0.15 Co	½HT	2 hr. at 575 F. in purified nitrogen	0.032	Parallel	170 400	79 900	98 800	19 200 000	4.4	C36.0	33 700	0.20
A5-45-XIII	2.00 Be, 0.35 Ni	½HT	2 hr. at 600 F. in muffle furnace	0.032	Parallel	200 900	106 000	119 400	19 600 000	3.0	C41.3	42 500^c	0.21
					45 deg.	197 200	104 900	118 300	18 400 000	2.5	C41.3	....	....
C4-45-XIII	2.00 Be, 0.35 Ni	½HT	2 hr. at 600 F. in muffle furnace	0.032	Parallel	180 400	89 700	108 600	19 300 000	6.7	C37.4	44 000^c	0.24
					45 deg.	170 500	88 700	107 700	18 900 000	7.2	C37.4	....	....
E3-45-XIII	2.00 Be, 0.15 Co	½HT	2 hr. at 600 F. in muffle furnace	0.032	Parallel	163 900	74 500	84 100	18 500 000	5.2	C33.4	41 000^e	0.25
					45 deg.	158 400	69 600	77 900	18 000 000	3.8	C33.4	....	....
A5-45-XIV	2.00 Be, 0.35 Ni	½HT	2 hr. at 600 F. in purified nitrogen	0.032	Parallel	....	....	....	....	....	C41.7	39 700^d	....
A5-45-IX	2.00 Be, 0.35 Ni	½HT	1 hr. at 600 F. in salt bath	0.032	Parallel	....	....	....	....	....	C41.1	38 500	0.20
A6-45-IX	2.00 Be, 0.35 Ni	HT	1 hr. at 600 F. in salt bath	0.032	Parallel	195 800	66 700	90 700	20 100 000	3.3	C40.5	....	....
A5-45-X	2.00 Be, 0.35 Ni	½HT	8 min. at 780 F. in salt bath	0.032	Parallel	....	....	....	....	....	C35.8	40 500	0.24^f
A6-45-X	2.00 Be, 0.35 Ni	HT	8 min. at 780 F. in salt bath	0.032	Parallel	166 800	68 200	88 800	20 200 000	7.8	C35.7	....	....

Continued on p. 746.

TABLE II (Concluded)

Designation	Nominal Composition, per cent	Condition	Precipitation-Hardening Treatment	Nominal Thickness, in.	Direction of Rolling with Respect to Length of Specimen	Tensile Strength, psi.	Proportional Limit, psi.	Yield Strength at 0.01 per cent Offset, psi.	Modulus of Elasticity, psi.	Elongation in 2 in., per cent	Rockwell Hardness	Fatigue Strength at 10^8 Cycles of Reversed Stress, psi.	Ratio of Fatigue Strength to Tensile Strength
1945 INVESTIGATION (*Continued*)													
A5-45-XI	2.00 Be, 0.35 Ni	½HT	45 min. at 525 F. in oil bath	0.032	Parallel	....	....	....	....	....	C32.2	39 200	0.27[f]
A6-45-XI	2.00 Be, 0.35 Ni	HT	45 min. at 525 F. in oil bath	0.032	Parallel	146 300	68 100	97 900	19 100 000	10.5	C30.4	....	....
A5-45-XV	2.00 Be, 0.35 Ni	½HT	2 hr. at 600 F. in muffle furnace	0.032	Parallel	....	....	....	....	....	C40.0	40 500[e]	0.21[f]
A6-45-XV	2.00 Be, 0.35 Ni	HT	2 hr. at 600 F. in muffle furnace	0.032	Parallel	195 500	66 700	106 300	19 600 000	3.5	C40.3	....	....
A6-45	2.00 Be, 0.35 Ni	H	None	0.032	Parallel	108 600	59 200	71 200	17 700 000	7.5	B96.4	37 000	0.34
					45 deg.	105 600	54 700	65 500	16 400 000	9.2	B96.4	....	
C5-45	2.00 Be, 0.15 Co	H	None	0.032	Parallel	104 600	61 000	70 500	16 400 000	5.0	B96.1	35 000	0.34
					45 deg.	105 600	52 500	67 000	17 800 000	6.8	B96.1	....	
E4-45	2.00 Be, 0.35 Ni	H	None	0.032	Parallel	113 600	61 100	74 800	17 600 000	4.4	B98.6	36 000	0.32
					45 deg.	111 200	62 000	74 400	17 100 000	5.4	B98.6	....	
E7-45 C	2.00 Be, 0.35 Ni	H	None	0.032	Parallel	109 900	41 400	62 900	17 600 000	5.9	B97.9	39 000	0.36
A6-45-II	2.00 Be, 0.35 Ni	HT	2 hr. at 600 F. in purified nitrogen	0.032	Parallel	199 300	92 400	113 100	19 500 000	2.9	C41.1	41 800	0.21
C5-45-II	2.00 Be, 0.15 Co	HT	2 hr. at 600 F. in purified nitrogen	0.032	Parallel	180 500	53 800	86 200	19 200 000	4.6	C37.7	41 000	0.23
E4-45-II	2.00 Be, 0.35 Ni	HT	2 hr. at 600 F. in purified nitrogen	0.032	Parallel	188 900	86 500	106 400	18 900 000	3.7	C40.0	38 900	0.21
E7-45 C-XVII	2.00 Be, 0.35 Ni	HT	2 hr. at 600 F. in muffle furnace	0.032	Parallel	191 500	65 600	95 700	19 400 000	3.1	C40.7	41 000	0.22
C5-45-IV	2.00 Be, 0.15 Co	HT	2 hr. at 575 F. in purified nitrogen	0.032	Parallel	182 400	82 800	105 800	19 000 000	4.6	C38.6	38 000	0.21
A6-45-XIII	2.00 Be, 0.35 Ni	HT	2 hr. at 600 F. in muffle furnace	0.032	Parallel	196 700	80 200	90 700	19 900 000	3.0	C40.5	47 500[c]	0.24
					45 deg.	193 300	77 700	94 600	19 300 000	4.0	C40.5	....	
E4-45-XIII	2.00 Be, 0.15 Co	HT	2 hr. at 600 F. in muffle furnace	0.032	Parallel	145 500	63 800	72 100	19 800 000	7.4	C30.2	51 000[c]	0.35
					45 deg.	147 200	63 400	70 700	19 000 000	8.0	C30.2	....	
C5-45-XIII	2.00 Be, 0.35 Ni	HT	2 hr. at 600 F. in muffle furnace	0.032	Parallel	186 400	76 700	93 300	19 700 000	4.5	C39.0	45 000[c]	0.24
					45 deg.	183 600	78 100	95 900	19 700 000	4.0	C39.0	....	
A6-45-XVIII	2.00 Be, 0.35 Ni	HT	20 min. at 660 F. in salt bath	0.032	Parallel	192 300	....	....	....	3.5	C39.9	43 000	0.22
A6-45-XIX	2.00 Be, 0.35 Ni	HT	20 min. at 700 F. in salt bath	0.032	Parallel	190 100	....	....	....	5.0	C38.2	48 000	0.25
Requirements of A.S.T.M. Designations B 120 – 41 T and B 194 – 45 T													
		A	None	....	Parallel	80 000 max.	....	....	....	35.0 min.	B80 max.	....	
		½H	None	....	Parallel	80 000 min.	....	....	....	5.0 min.	B82 min.	....	
		H	None	....	Parallel	95 000 min.	....	....	....	2.0 min.	B93 min.	....	
		AT	3 hr. at 600 F.	....	Parallel	150 000 min.	....	....	....	5.0 min.	C33 min.	....	
		½HT	2 hr. at 600 F.	....	Parallel	170 000 min.	....	....	....	2.0 min.	C37 min.	....	
		HT	2 hr. at 600 F.	....	Parallel	180 000 min.	....	....	....	1.0 min.	C39 min.	....	

[a] Experimental alloy.
[b] Yield strength at 0.005 per cent offset.
[c] Fatigue specimens were polished with No. 400 Aloxite paper prior to test.
[d] Fatigue specimens were polished on buffing wheel.
[e] Fatigue specimens were pickled.
[f] Ratio based upon tensile strength of condition HT material which is assumed to be the same as that of Condition ½HT

were made on two commercial lots of the cobalt-bearing ternary alloy aged in a salt bath for 2 hr. at 600 F. as recommended by the supplier.

3. Studies made in 1942 to determine the effect of higher nickel contents (1 to 2 per cent) on the fatigue characteristics and the tensile properties. The tests were made on the standard binary alloy and on four experimental ternary alloys using specimens aged in a salt bath for 1 hr. at 600 F. as recommended by the supplier.

4. Studies made in 1945 to determine the fatigue characteristics of the standard commercial alloys as supplied by three of the principal producers.

These materials were purchased in three tempers: conditions A, ½H, and H, in the spring of 1944 on the open market in accordance with the requirements of Tentative Specifications for Beryllium-Copper Alloy Rods, Bars, Sheet, Strip, and Wire (B 120 – 41 T).[3] Tests were made on samples "as received," after precipitation-hardening at 600 F. in accordance with the acceptance test specified in Tentative Specifications B for Beryllium-Copper Alloy Strip (194 – 45 T),[4] and after various other precipitation-hardening treatments. The effects of pickling, buffing, and polishing were also studied. Three check lots of the ternary nickel-bearing alloys were included to verify previous test results.

TESTING PROCEDURE AND TEST DATA

The chemical analyses of the various alloys, as determined in accordance with approved methods of wet analysis for copper-base alloys are given in Table I. In this table, all material from the same source is identified by a letter and a single digit followed by two numbers which indicate the year in which the material was tested.

The tensile strength of the material was determined in accordance with the Standard Methods of Tension Testing of Metallic Materials (E 8 – 42),[5] using cross-milled specimens similar to those shown in Fig. 2 of that method. The proportional limit, yield strength at 0.01 per cent offset, elastic modulus, and per cent elongation in 2 in. were determined on these same specimens. The tension tests were made on a 20,000-lb. Amsler universal testing machine. This machine was accurate to ±1 per cent at any load. The scale used was such that fracture of the test specimens occurred at one half to three quarters of the full scale reading. For the determination of the elastic properties of materials studied prior to 1944, a Riehlé-Sayre extensometer reading to 0.00002 in. was used in connection with a drum recorder. For some of the tests made after 1944, a Templin extensometer reading to 0.00002 in. was used with a Baldwin-Southwark automatic recorder. The percentage elongation was determined with dividers to the nearest 0.5 per cent. In general, five tension test specimens were used.

The results of the various physical tests, including Rockwell hardness data are given in Table II. The Roman numerals, used as part of the designation, refer to the various heat treatments listed in the fourth column of this table and in the bar-graph summary, Fig. 22.

The Rockwell hardness tests were made in accordance with the Standard Methods of Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (E 18 – 42)[6] on the ends of the tension test specimens. The hardness-time curves shown in Fig. 1 were determined from observations made on 1-in. square specimens, precipitation-hardened in a muffle furnace in an atmosphere of purified nitrogen. Since these specimens were not heat-treated at the

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[3] 1942 Book of A.S.T.M. Standards, Part I, p. 1259. (Superseded by Specifications B 194, B 195, B 196, and B 197.)

[4] 1945 Supplement to Book of A.S.T.M. Standards, Part I, p. 240.

[5] 1944 Book of A.S.T.M. Standards, Part I, p. 962.

[6] 1944 Book of A.S.T.M. Standards, Part I, p. 979.

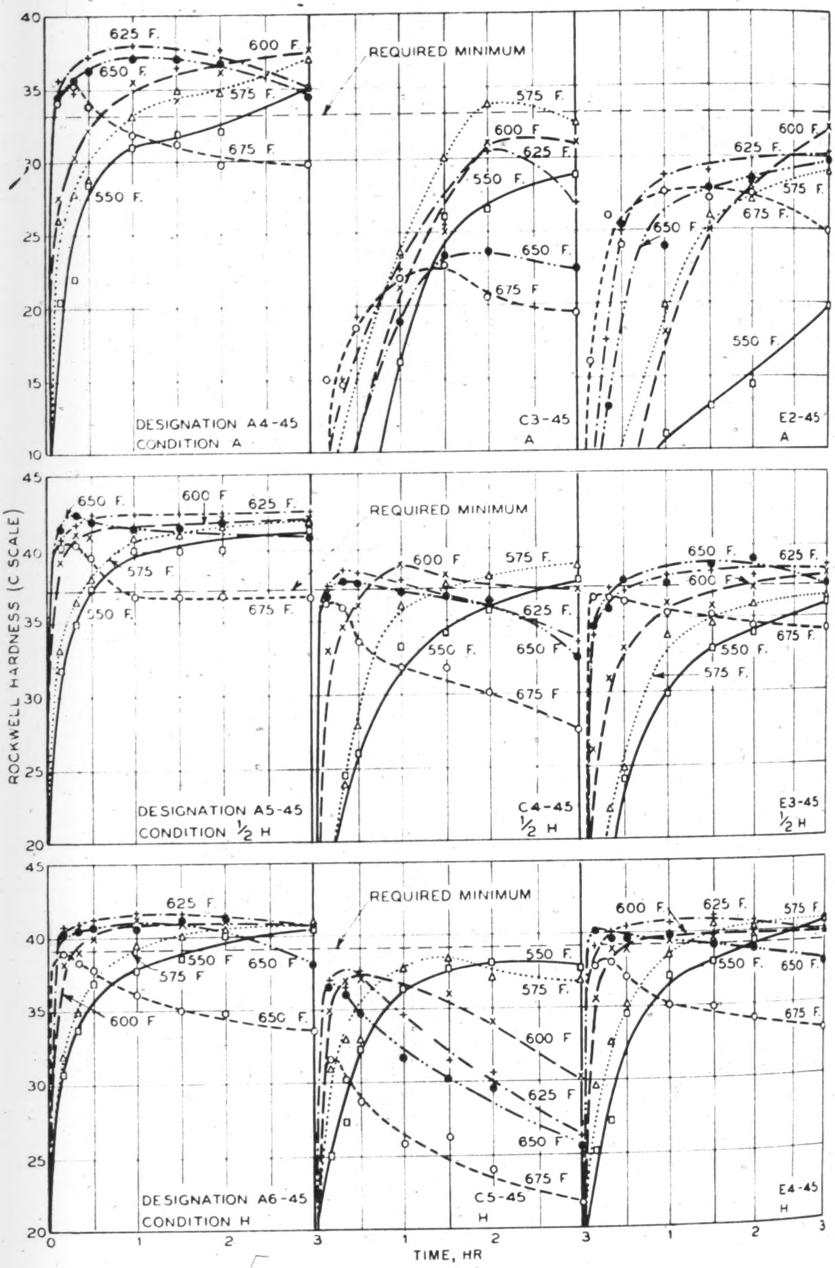

FIG. 1.—Precipitation-Hardening Characteristics of Beryllium-Copper Strip.

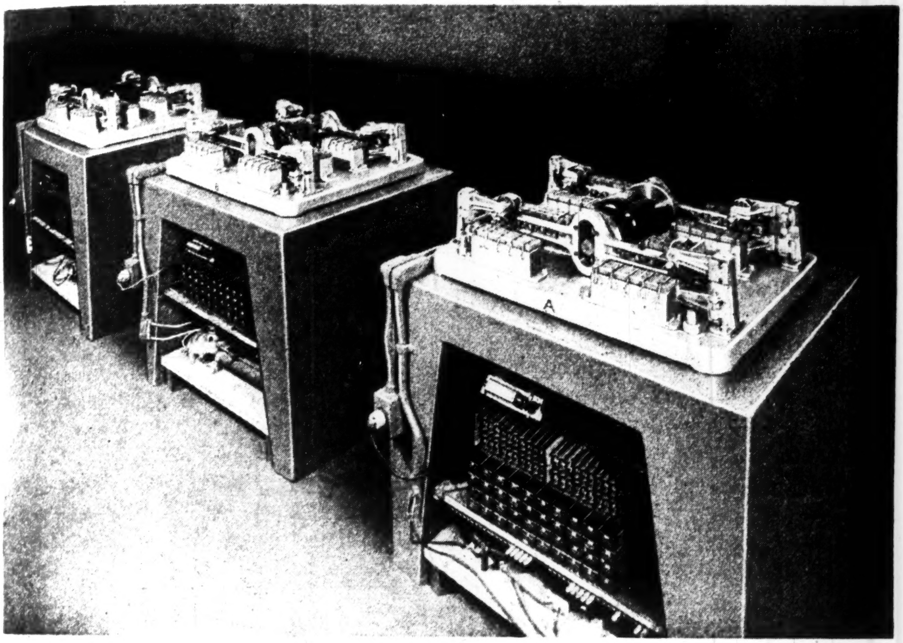

FIG. 2.—Sheet Metal Fatigue Machines.

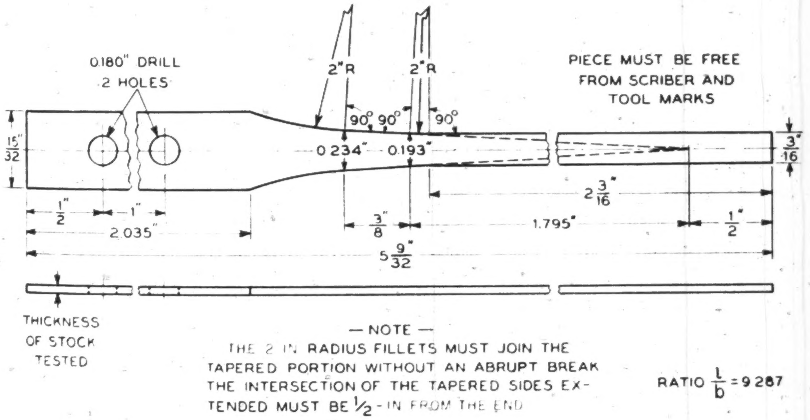
0.180" DRILL
2 HOLES
2"R
2"R
PIECE MUST BE FREE
FROM SCRIBER AND
TOOL MARKS
90°
90°
90°
15/32"
0.234"
0.193"
3/16"
2 3/16"
1/2"
1"
2.035"
3/8"
1.795"
1/2"
5 9/32"
THICKNESS
OF STOCK
TESTED
— NOTE —
THE 2 IN RADIUS FILLETS MUST JOIN THE
TAPERED PORTION WITHOUT AN ABRUPT BREAK
THE INTERSECTION OF THE TAPERED SIDES EX-
TENDED MUST BE ½-IN FROM THE END
RATIO l/b = 9.287

FIG. 3.—Fatigue Test Specimen.

same time as the tension specimens, there may be slight differences between the hardness values shown in Fig. 1 and those shown in Table II for similar heat-treatment of a given material.

The fatigue strengths[7] of the various materials were determined on the multi-specimen constant strain, flexure type fatigue machines shown in Fig. 2. These machines were developed by the Bell Telephone Laboratories and have been described by Townsend and Greenall (6). The details of test, subsequently described by Greenall and Gohn (4) were followed in these studies using cross-milled specimens having the dimensions shown in Fig. 3. Precipitation-hardened specimens were heat-treated after cross-milling. All of the tests were made at a speed of 750 to 900 rpm. at a temperature of 80 to 85 F. During test, the $\frac{3}{8}$-in. tapered section (Fig. 3) is subjected to a uniform bending stress when the free end of the specimen is deflected by the reciprocating arm of the testing machine. The deflections were checked periodically by dynamic methods to insure that no change in stress had occurred. Maximum bending stresses were computed as described in the previous paper (4). In general, five specimens were tested at each deflection, and the results obtained are precise to at least ±3 per cent.

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[7] For brevity, throughout this paper, the term "fatigue strength" is used hereafter rather than the full designation "fatigue strength at 10^8 cycles of reversed stress."

The fatigue strengths and the ratios between fatigue and tensile strength are given in Table II for the various lots of beryllium-copper strip. The *S-N* diagrams, from which the endurance limits may be derived, are shown in Figs. 4 to 16, inclusive.

The metallographic studies were made at magnifications of 500 on polished and etched sections taken from the fatigue specimens. These sections were normal to the surface and parallel to the direction of rolling; they were prepared as described in the Tentative Methods of Preparation of Metallographic Specimens (E 3 - 44 T)[8] and etched with the solutions indicated on the photomicrographs, Figs. 17 to 20.

DISCUSSION OF DATA

1936 Investigation:

Greenall and Gohn (4) made no detailed analysis of the effect of age hardening or cold working on the fatigue strength of the beryllium-copper alloys. However, a further study of their data for the experimental alloys shows very little difference in the fatigue strength of condition AT, $\frac{1}{2}$HT or HT material. In the case of one of the commercial alloys, an increase in fatigue strength of 15 to 30 per cent was obtained by heat-treating condition $\frac{1}{2}$H material for 1 or 2 hr. at 525 F. Condition H material, so heat-treated, showed a gain of over 30 per cent in fatigue strength. However, the maximum fatigue strength obtained was only 36,000 psi. While the tensile properties resulting from this heat treatment met the requirements now prescribed in Tentative Specifications B 194 - 45 T,[4] in the light of our present knowledge these specimens were undoubtedly underaged.

1941 Investigation:

In the studies made in 1941, the specified tensile and Rockwell hardness requirements were met by both the nominal 2.00 and the 1.85 per cent beryllium-cobalt alloys, after heat treatment in a salt bath at 600 F. As would be expected, the alloy of lower beryllium content did not reach as high a tensile strength or Rockwell hardness level as did the alloy of higher beryllium content, although the elongation was slightly better. However, despite the differences in tensile strength and elongation, there was no significant difference in the fatigue strength of the corresponding tempers of the two alloys (Fig. 4). While the temperature at which these materials were heat-treated reflects present commercial practices, the time (3 hr. for condition A and 2 hr. for conditions $\frac{1}{2}$H and H material) was double that usually employed for salt-bath treatments.

1942 Investigation:

The 1942 investigation was primarily concerned with experimental alloys in

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[8] 1944 Book of A.S.T.M. Standards, Part I, p. 1884.

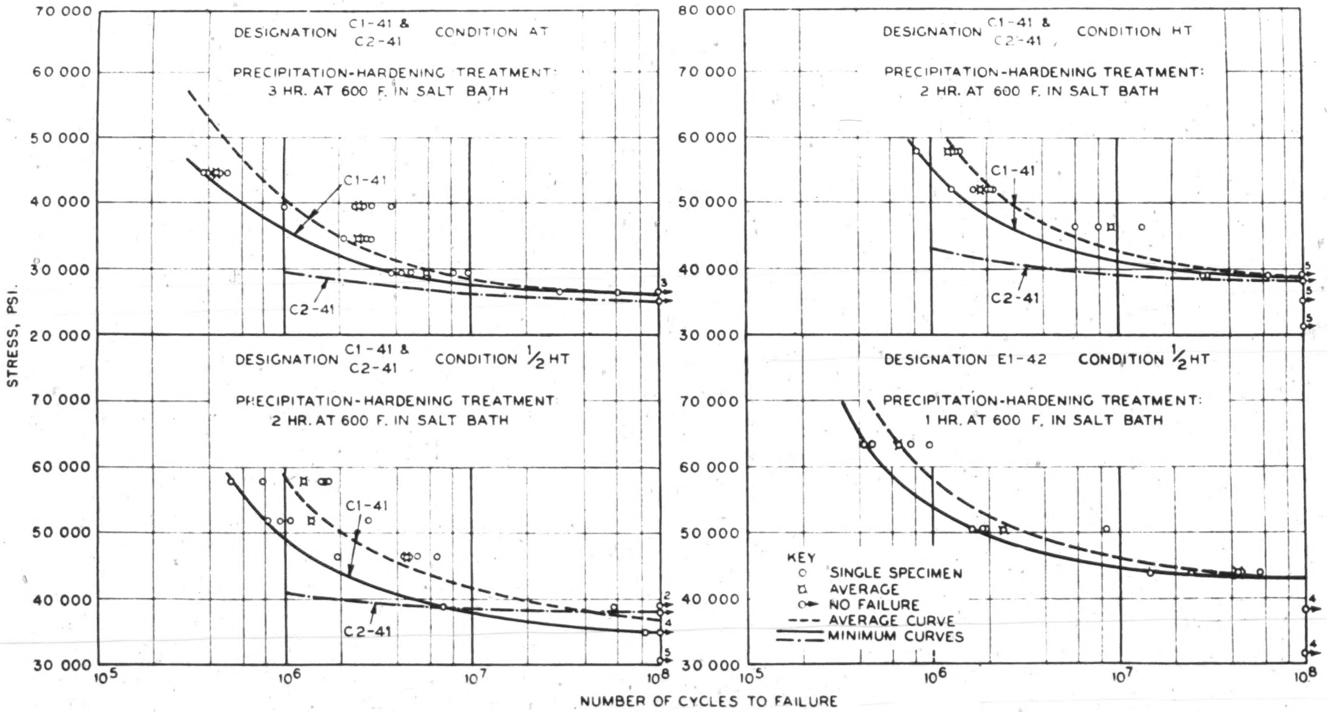


FIG. 4.—Fatigue Characteristics of Beryllium-Copper Strip.

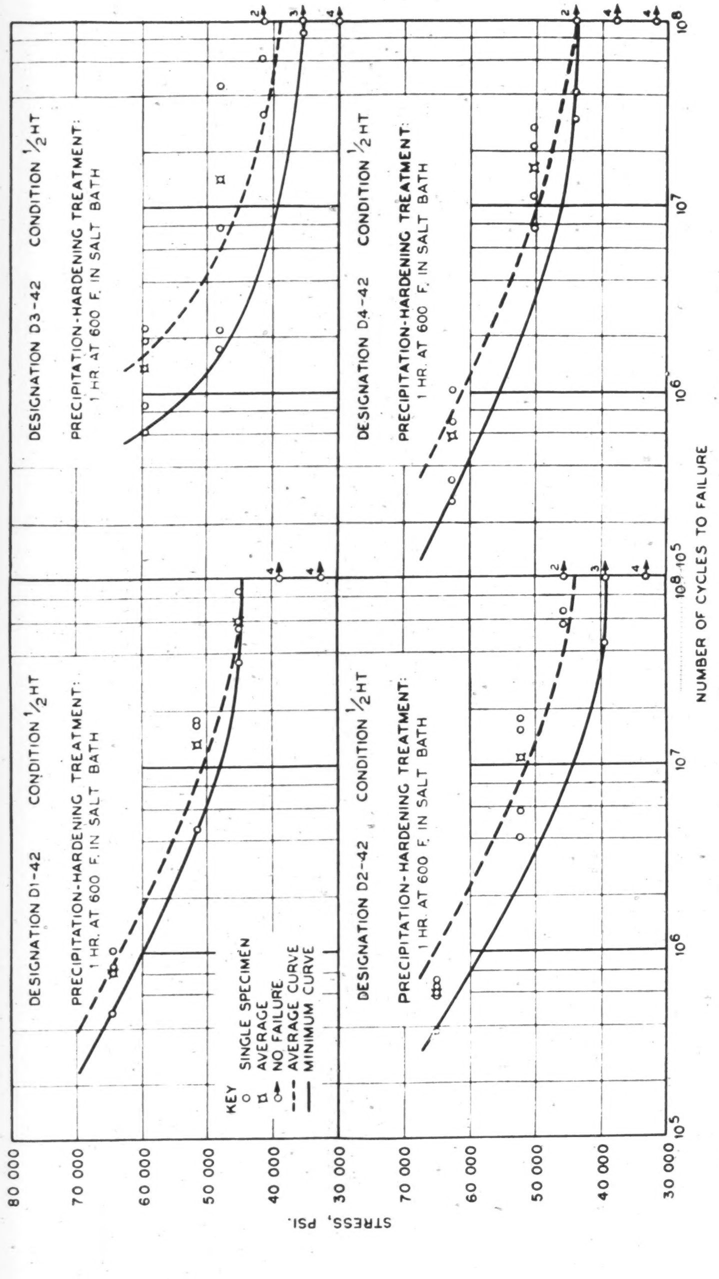


FIG. 5.—Fatigue Characteristics of Beryllium-Copper Strip.

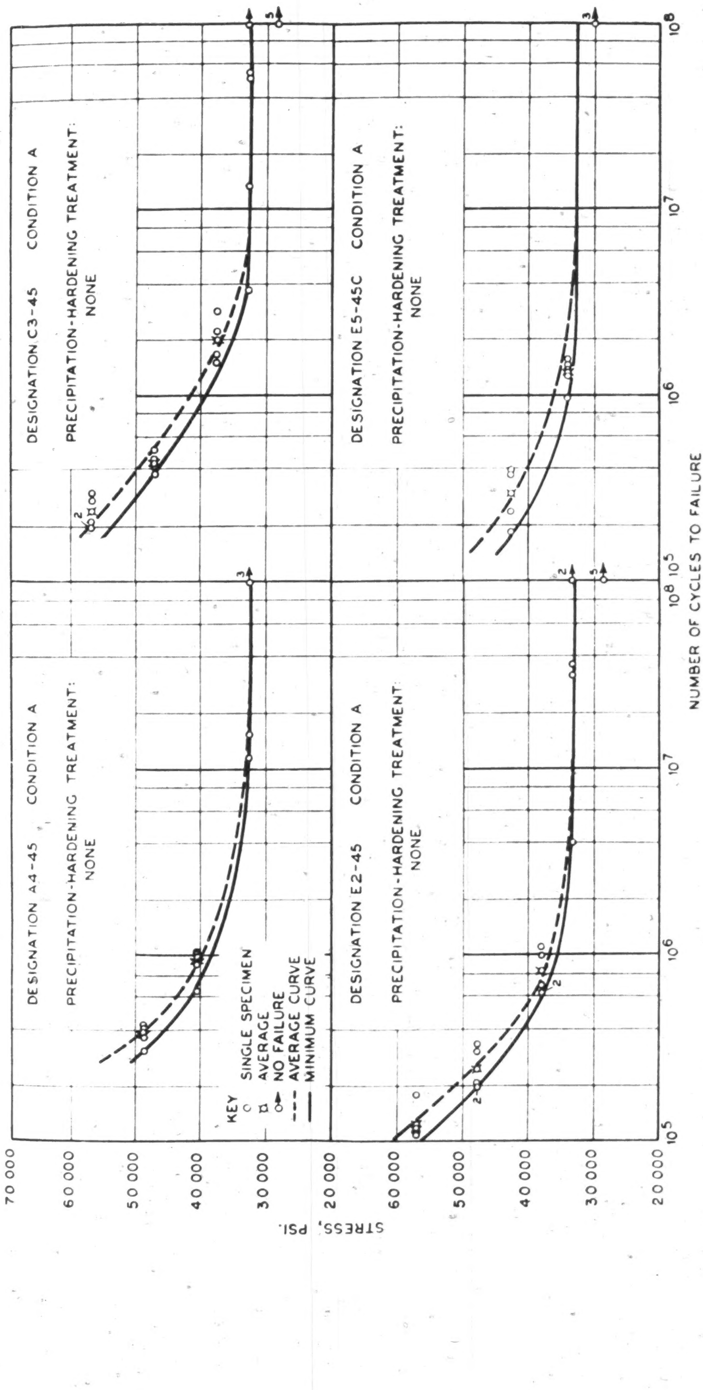


FIG. 6.—Fatigue Characteristics of Beryllium-Copper Strip.

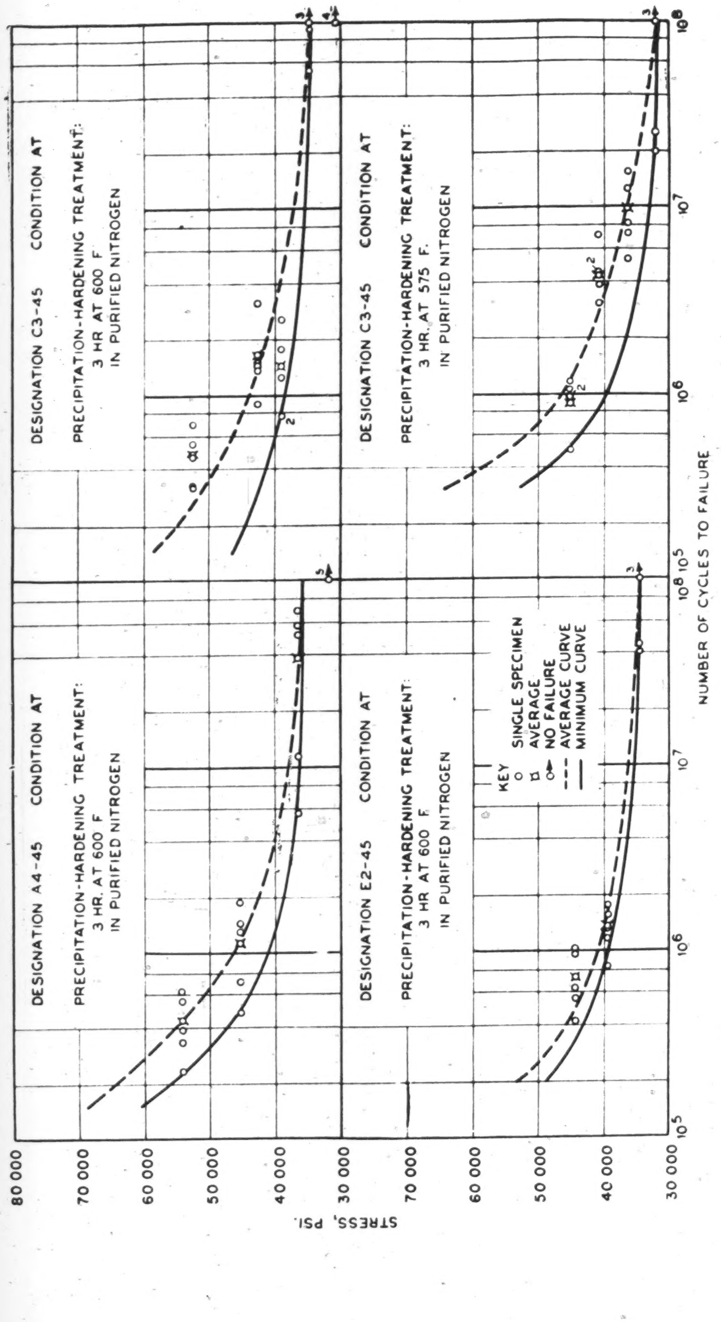


Fig. 7.—Fatigue Characteristics of Beryllium-Copper Strip.

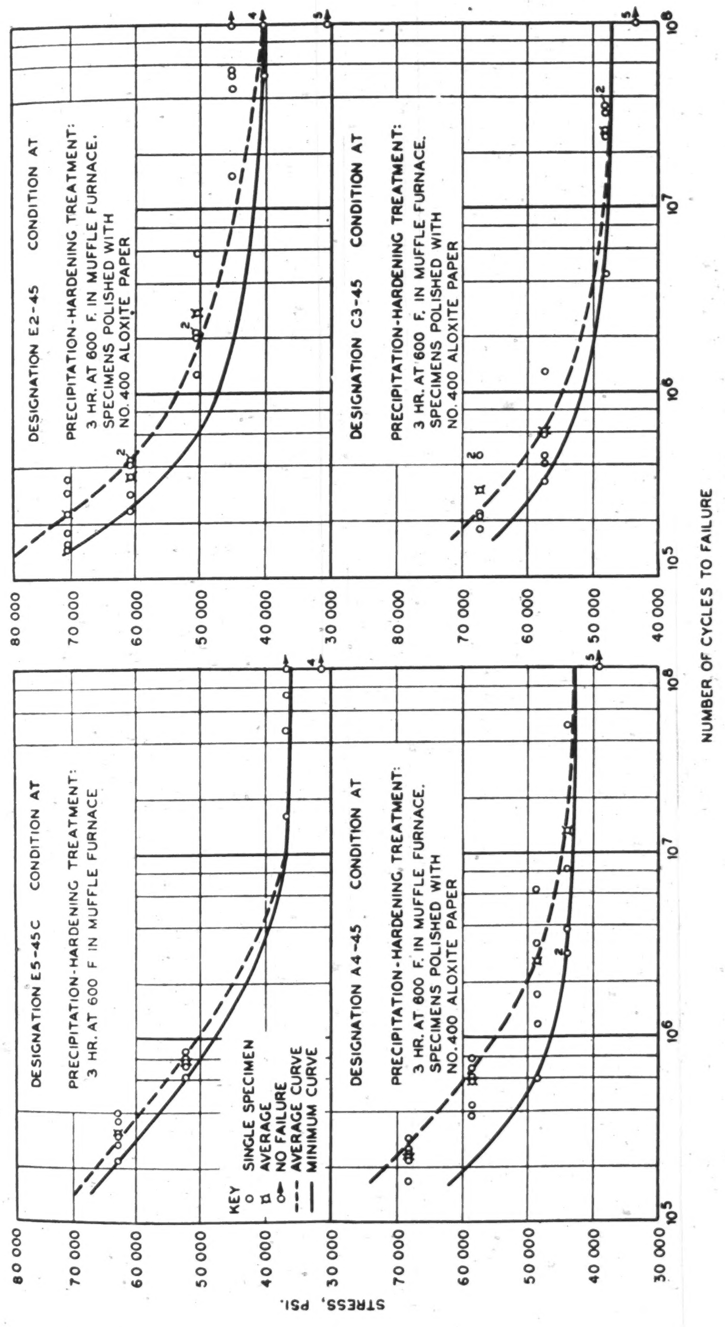


FIG. 8.—Fatigue Characteristics of Beryllium-Copper Strip.

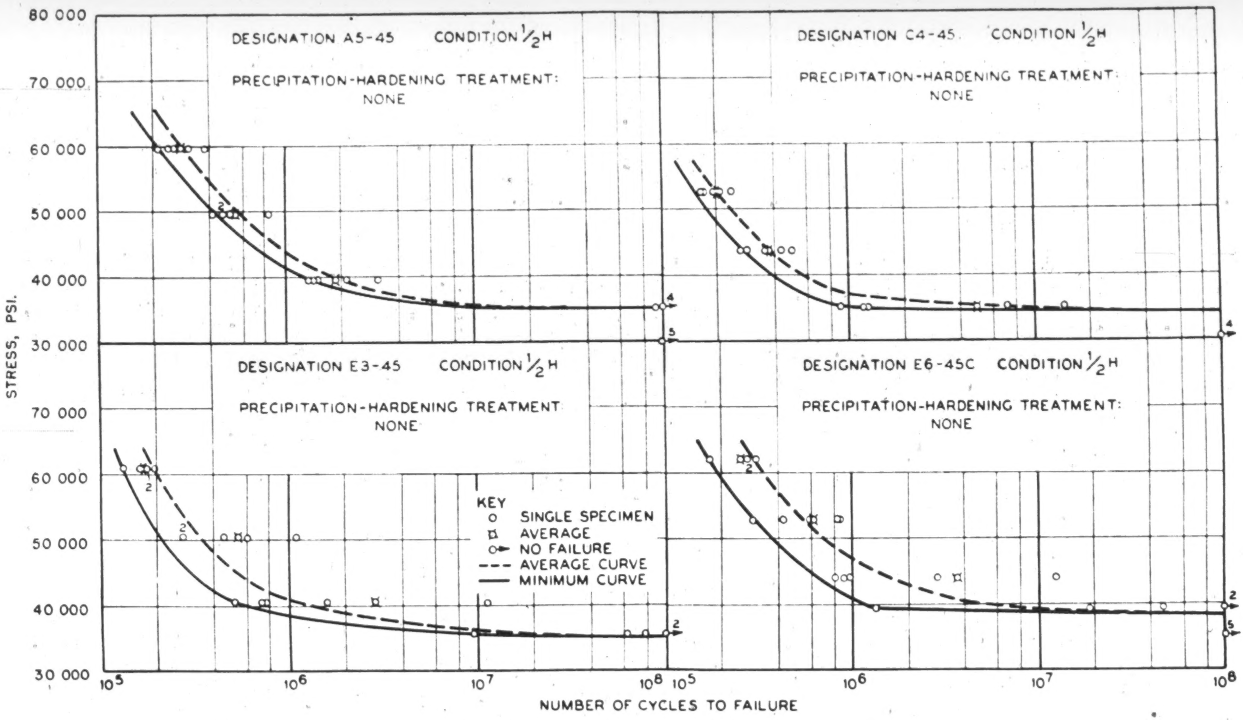


FIG. 9.—Fatigue Characteristics of Beryllium-Copper Strip.

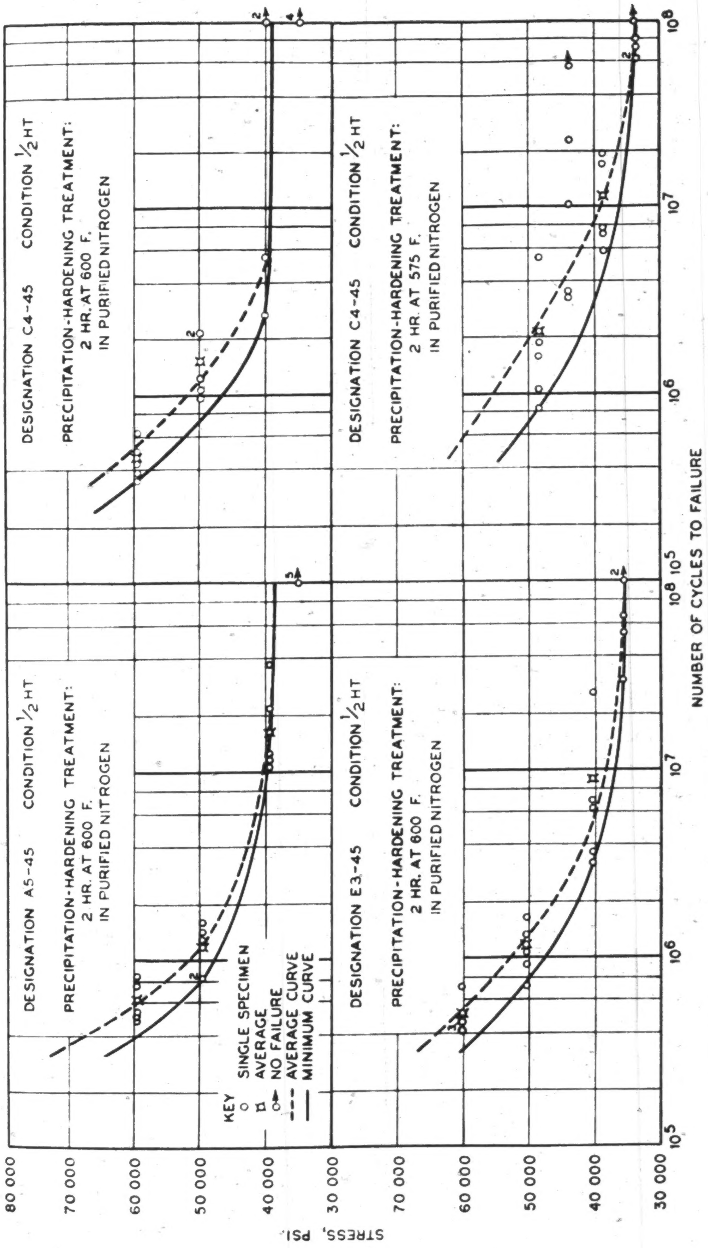


FIG. 10.—Fatigue Characteristics of Beryllium-Copper Strip.

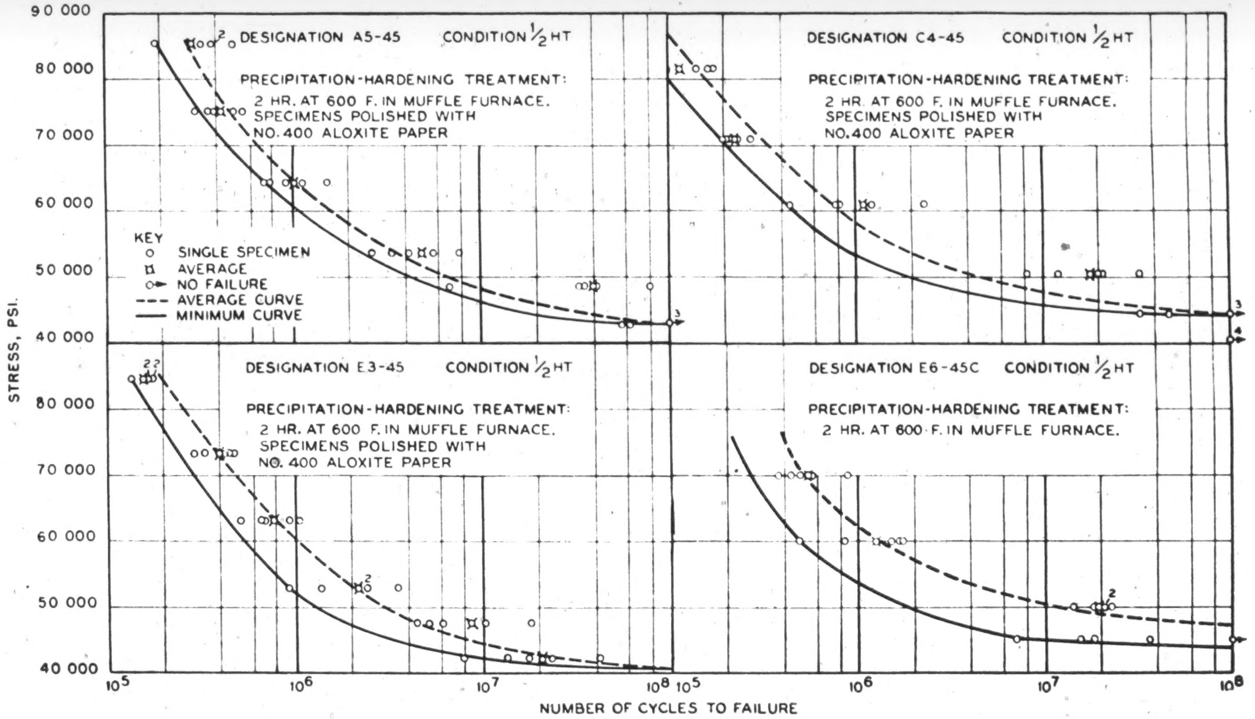


FIG. 11.—Fatigue Characteristics of Beryllium-Copper Strip.

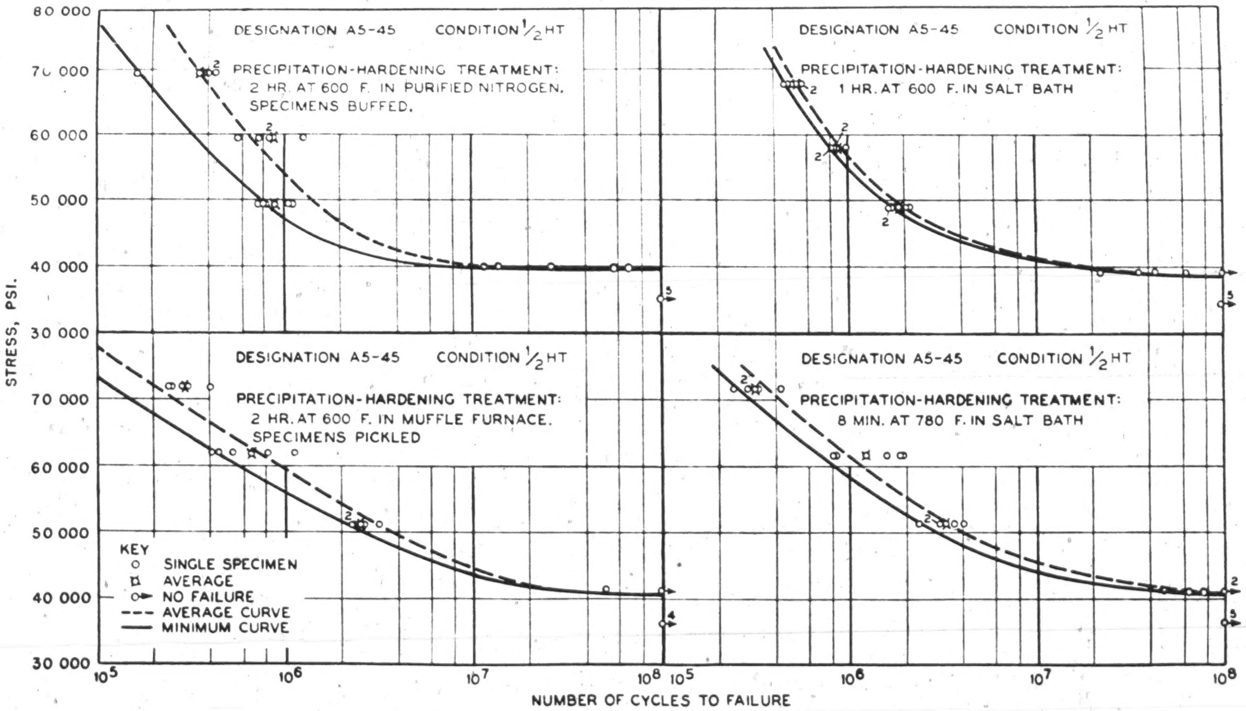


Fig. 12.—Fatigue Characteristics of Beryllium-Copper Strip.

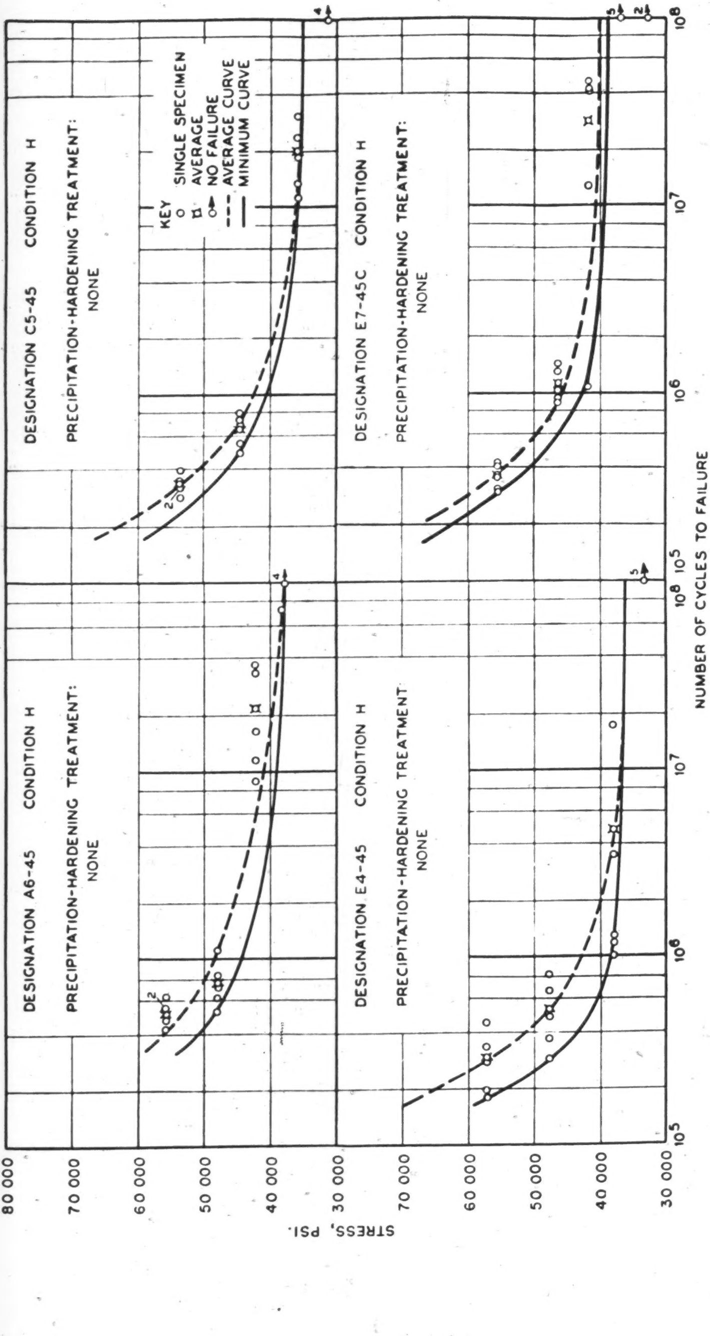


FIG. 13.—Fatigue Characteristics of Beryllium-Copper Strip.

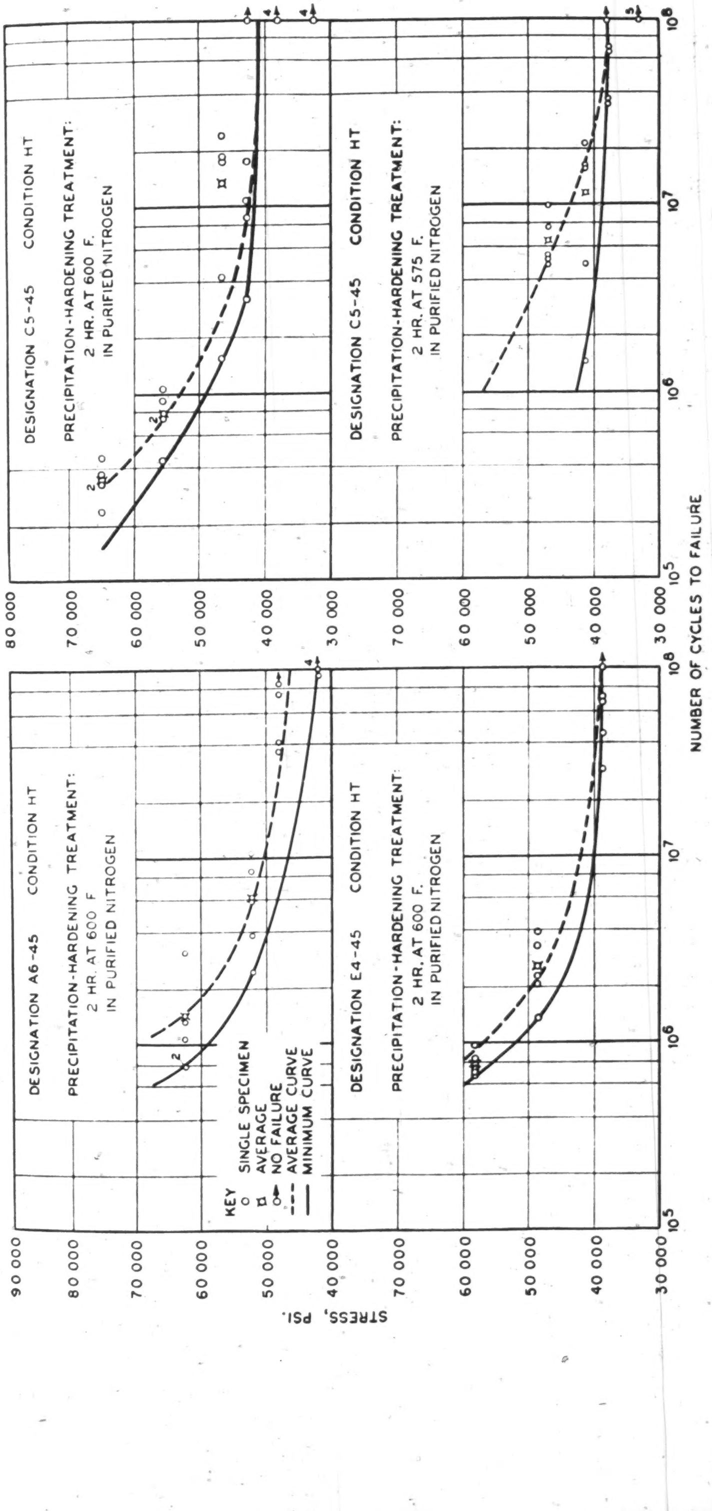


Fig. 14.—Fatigue Characteristics of Beryllium-Copper Strip.

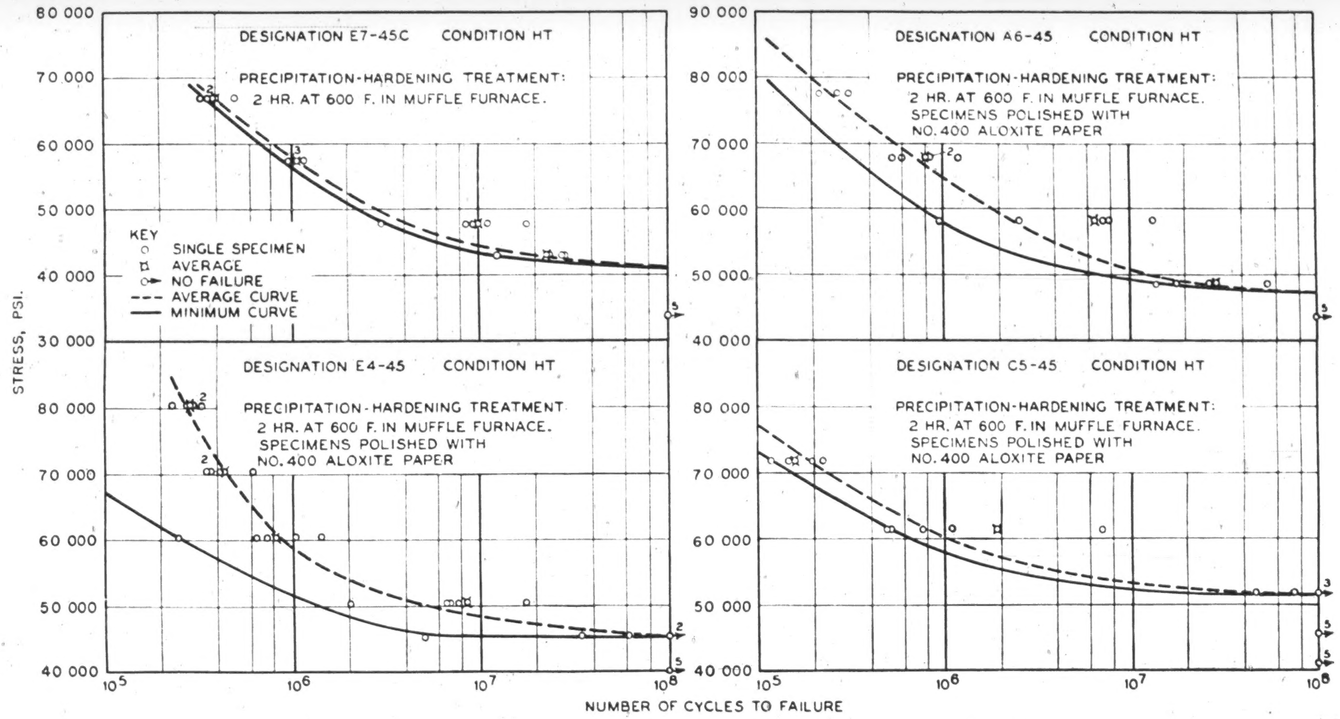


FIG. 15.—Fatigue Characteristics of Beryllium-Copper Strip.

which the nickel content was increased to values above the 0.50 per cent maximum permitted in Tentative Specifications B 120 - 41 T.[3] The studies included alloys in which 1 and 2 per cent of nickel were added to the 2.00 and 2.20 per cent binary beryllium-copper alloys. The alloys were tested only in the $\frac{1}{2}$HT condition after heat treatment in a salt bath for 1 hr. at 600 F. The physical properties of these alloys, along with those of a similarly treated binary control alloy containing 2.25 per cent beryllium, are given in Table II under the subheading 1942 Investigation.

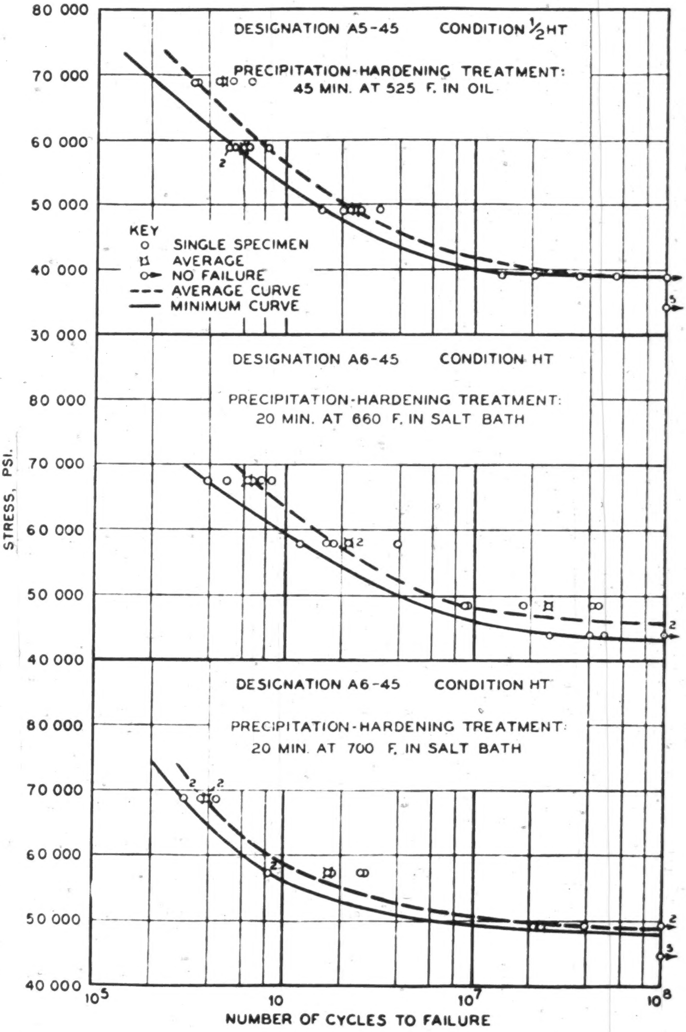


Fig. 16.—Fatigue Characteristics of Beryllium-Copper Strip.

Interesting data were obtained which reflect the combined effect of nickel and beryllium additions to copper. Whereas the tensile strength of the heat-treated 1 and 2 per cent nickel-bearing alloys was less than that of the binary alloy, the proportional limit and yield strength values were greater. In fact, the yield strength values of the nickel-bearing 2.20 per cent beryllium alloys were among the highest of any of the alloys studied by the authors. The decrease in tensile strength of the precipitation-hardened alloys with increased nickel content is consistent with the generally accepted belief that, through the formation of a nickel-beryllium compound, additions of nickel in large quantities deplete the beryllium available for hardening.

No relationship was found between fatigue strength and the variation in nickel and beryllium contents.

It may also be noted from the *S-N* diagrams in Fig. 5 that there was considerable spread between the average and the minimum fatigue values for the alloys studied in the 1942 investigation. This has been found to be typical of experimental alloys and may be due to lack of homogeneity of material manufactured in small-scale lots. In this connection it is of interest to note that a more consistent grouping of test data was obtained for the control alloy and for the commercial alloys reported in the 1945 investigation.

1945 Investigation:

The 1945 investigation was concerned primarily with nine commercial ternary alloys, containing either nickel or cobalt as the additive element. The materials were purchased in accordance with the requirements of Tentative Specifications B 120 - 41 T.[3] Later, three additional alloys were obtained to check the fatigue characteristics of material obtained from one source. Of particular interest were the physical properties of the materials "as received" and those developed following the heat treatment specified in the acceptance test in Tentative Specifications B 194 - 45 T.[4]

Chemical Composition:

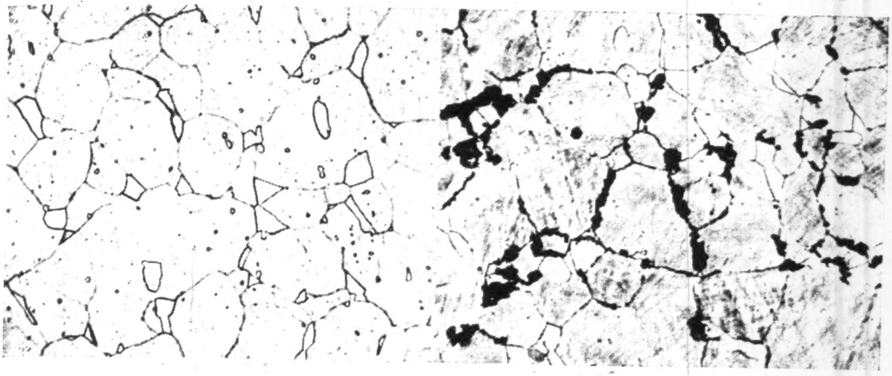
Chemical analyses of the twelve alloys included in this investigation are given in Table I. It should be noted that for material from different suppliers there was a considerable range both in beryllium content (approximately 1.80 to 2.20 per cent) and also in the nickel and the cobalt additions. It should likewise be noted that not all of the compositions are within the limits specified for beryllium in Tentative Specifications B 120 - 41 T,[3] or for nickel plus cobalt plus iron specified in the new Tentative Specifications B 194 - 45 T.[4] Despite these differences, all of the alloys could be hardened to the specified tensile requirements.

Microstructure and Hardening Characteristics:

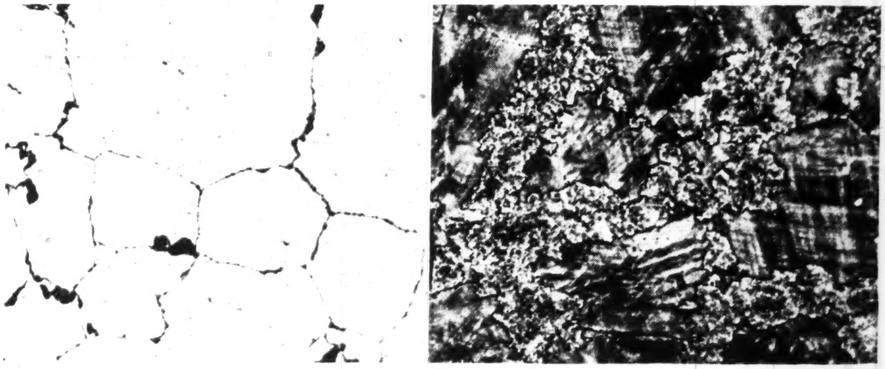
As may be seen from the photomicrographs, Figs. 17 to 20, all the alloys had a relatively small grain size, the average grain being less than 0.040 mm. The material from supplier C had a grain size approximately twice that of material from suppliers A and E, and probably this is associated with the smaller amount of additive element present.

It was noted that the materials with the highest beryllium content—those obtained from supplier A—had a considerable quantity of uniformly distributed beta constituent present. This beta was nearly or completely absent in the materials of lower beryllium content received from suppliers C and E. While there has been considerable discussion regarding the harmful effect of the presence of such beta constituent, in these studies no deleterious effect on fatigue strength was noticed. However, in extremely thin stock, the presence of the beta constituent may affect the processing and the forming characteristics.

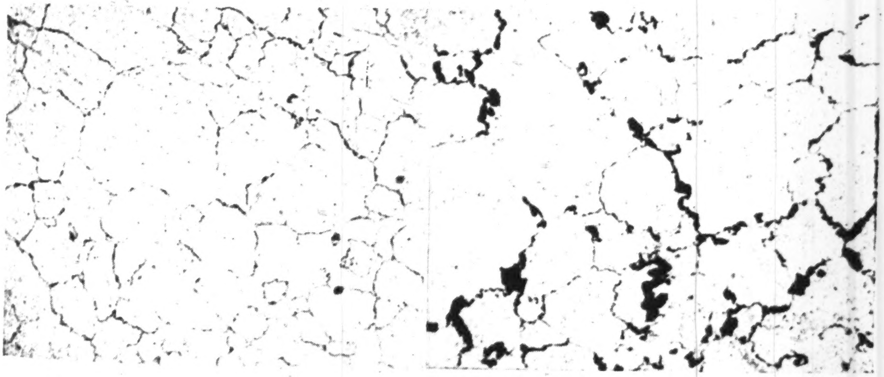
One alloy (C3-45 of Fig. 17) did show



Condition A Designation A4-45 Condition AT



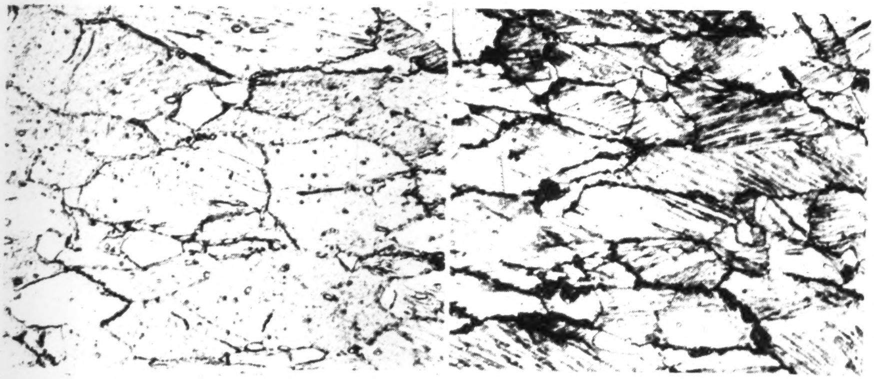
Condition A Designation C3-45 Condition AT



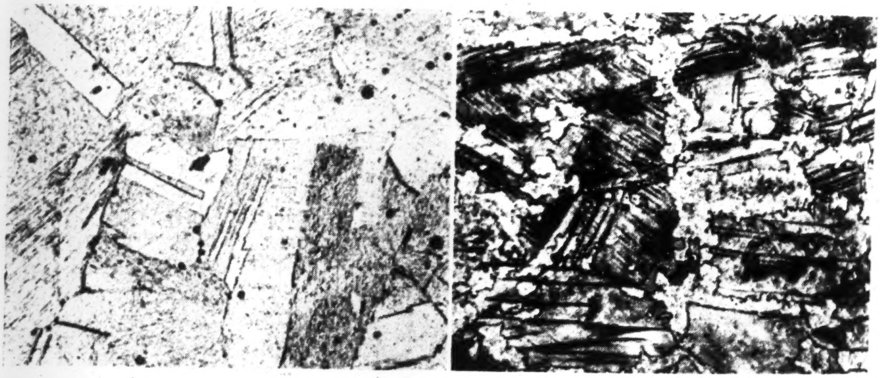
Condition A Designation E2-45 Condition AT

FIG. 17.—Structure of Beryllium-Copper Strip (× 500)

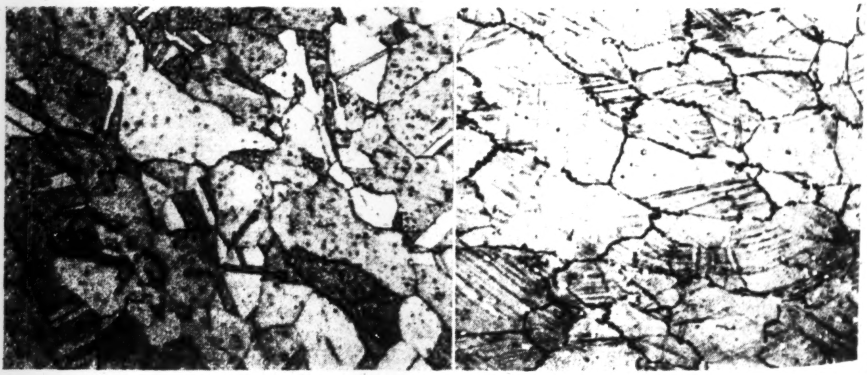
Etchant:
Condition A—Ammonium hydroxide and hydrogen peroxide.
Condition AT—Ammonium persulfate and ammonium hydroxide.
Precipitation-hardening treatment: 3 hr. at 600 F. in purified nitrogen.



Condition ½H Designation A5-45 Condition ½HT



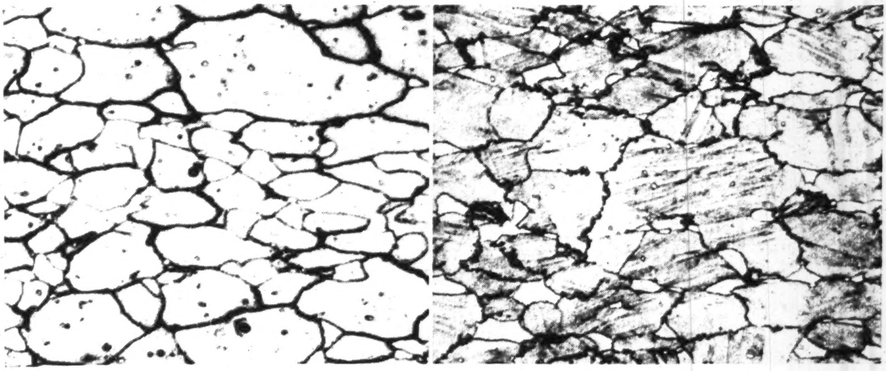
Condition ½H Designation C4-45 Condition ½HT



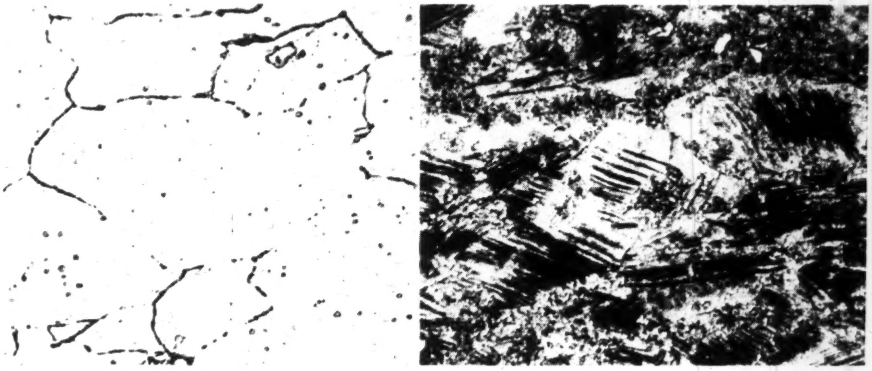
Condition ½H Designation E3-45 Condition ½HT

FIG. 18.—Structure of Beryllium-Copper Strip (× 500).

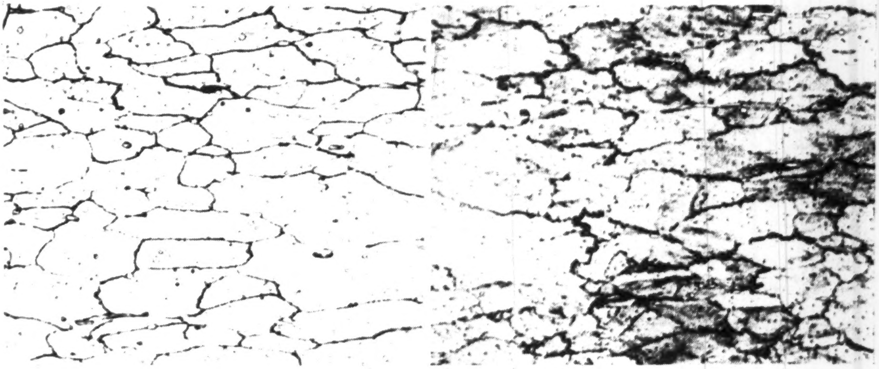
Etchant:
Condition ½H—Ammonium hydroxide and hydrogen peroxide.
Condition ½HT—Ammonium persulfate and ammonium hydroxide.
Precipitation-hardening treatment: 2 hr. at 600 F. in purified nitrogen.



Condition H Designation A6-45 Condition HT



Condition H Designation C5-45 Condition HT

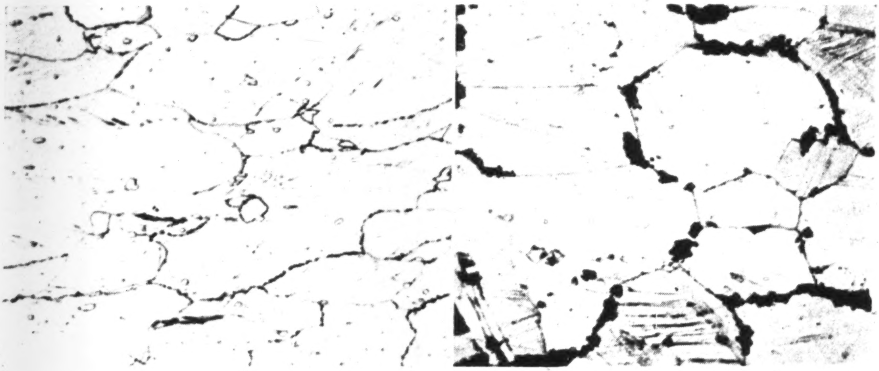


Condition H Designation E4-45 Condition HT

FIG. 19.—Structure of Beryllium-Copper Strip (× 500).

Etchant:
Condition H—Ammonium hydroxide and hydrogen peroxide.
Condition HT—Ammonium persulfate and ammonium hydroxide.
Precipitation-hardening treatment: 2 hr. at 600 F. in purified nitrogen.

in the unaged condition, some secondary beta or gamma constituent at the grain boundaries. This would indicate that the material had aged slightly during solution heat treatment. The formation of this secondary phase has abstracted some of the beryllium available for the subsequent formation of the primary gamma phase in the precipitation-hardening treatment. This, along with the initially low beryllium content, probably explains the erratic hardening characteristics of this material.



Designation A5-45 Condition ½HT Designation C4-45

FIG. 20.—Structure of Beryllium-Copper Strip (× 500).

Etchant:
Ammonium persulfate and hydrogen peroxide.
Precipitation-hardening treatment:
A5-45—45 min. at 525 F. in oil.
C4-45—2 hr. at 575 F. in purified nitrogen.

Although not shown in the photomicrographs included in this paper, a study was made of the various alloys to determine whether there was any difference in structure between specimens which, at the same stress, failed in fatigue after a maximum number of stress reversals and those which failed after a minimum number. These studies were made at two stresses, one high the other low, for each of the original nine alloys after heat treatment. Except in one case, no differences were noticed. The one exception showed some indication of segregation, but this could not be correlated with premature fatigue failure.

Referring now to the hardening characteristics, illustrated by the curves in Fig. 1, the interdependence of microstructure, chemical composition, and hardness may be noted. Material from supplier A, high in beryllium and containing some free beta, hardened rapidly and attained maximum physical properties in a shorter period of time than did the lower beryllium-content materials of suppliers C and E. Material low in beryllium had a narrow hardening range and the heat treatment was critical, both with respect to time and temperature. In some cases it was difficult to meet the specified minimum hardness requirements.

The relationship between the hardness values as determined on various scales is shown in Fig. 21. It can be seen that, with the exception of the G scale, the values actually obtained did not coincide with the conversion values suggested in Tentative Specifications B 194 – 45 T[4] or with those on the Wilson Mechanical Instrument Chart 38. As beryllium-copper is being specified more and more for spring applications, a need is indicated for additional studies on the hardness conversion values.

Since alloys from supplier C were found to develop maximum hardness as well as a more normal microstructure when heat-treated at temperatures below the 600 F. specified in the acceptance test, the study was extended to include specimens heat-treated at 575 F. The microstructure of one of the materials (alloy C4-45) heat-treated at these two temperatures is shown in Figs. 18 and 20. These tests are discussed after the analysis of the 600 F. aging studies.

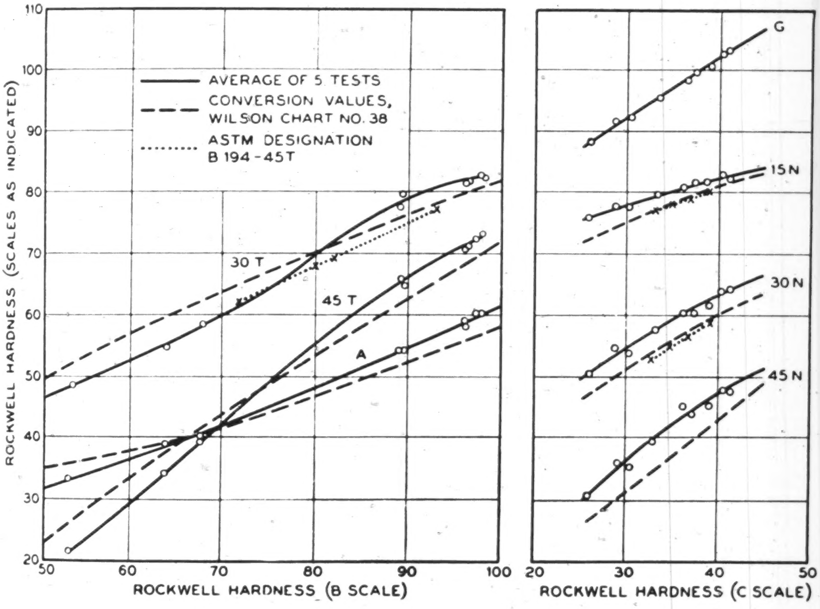


Fig. 21.—Rockwell Hardness Conversion Curves for Beryllium-Copper Strip.

Tensile Properties:

Regardless of source, all the materials both in the "as received" condition and after heat treatment at 600 F., met the requirements of Tentative Specifications B 120 – 41 T[3] with regard to tensile properties. After precipitation-hardening, the tensile strength was highest for the alloys with higher beryllium contents, as were the yield strength and proportional limit values. In general, while the yield strengths and proportional limits parallelled the tensile strength values, the variation was greater.

The physical properties for the rolled tempers in the "as received" condition were as good as or better than those obtained for the 5 per cent tin bronzes rolled to hard and spring tempers. After heat treatment, these values were materially increased, with a corresponding reduction in elongation. However, the low elongation and small spread between the tensile and the yield strength make it difficult to use beryllium-copper springs in applications requiring adjustment after assembly. The data in Table II show that by deviating from the standard heat treatment it is possible to obtain suitable fatigue properties, a greater spread between tensile and yield strength, and a sufficiently high elongation to permit the use of beryllium-copper

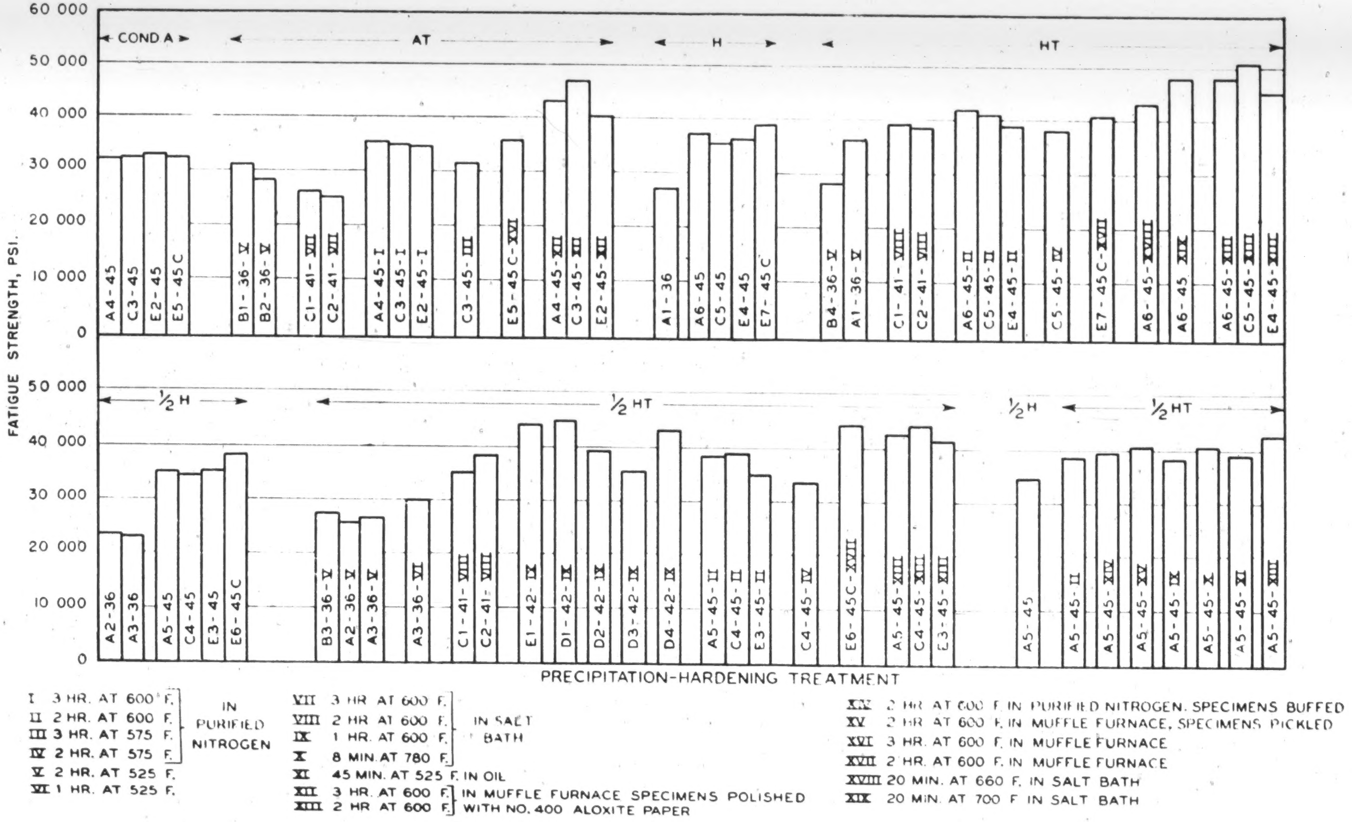


FIG. 22.—Fatigue Strength of Beryllium-Copper Strip.

in many applications where subsequent adjustment is required.

Further study of the data in Table II shows that, while there is some variation in the physical properties depending upon whether the specimens are taken parallel or at 45 deg. to the direction of rolling, this difference is slight and inconsistent. There was a marked difference in the modulus of elasticity for material in the "as received" condition and after heat treatment. The average value for the unaged material was found to be 17,500,000 psi.; after heat treatment, this value increased to 19,300,000 psi.

TABLE III.—EFFECT OF COLD-ROLLING AND PRECIPITATION-HARDENING ON FATIGUE STRENGTH.

Designation	Fatigue Strength, psi.		Furnace Atmosphere	Precipitation Hardening	
	Condition A	Condition AT		Time, hr.	Temperature, deg. Fahr.
A4-45	32 000 (Fig. 6)	35 500 (Fig. 7)	Nitrogen	3	600
C3-45	32 500 (Fig. 6)	34 800 (Fig. 7)	Nitrogen	3	600
E2-45	33 000 (Fig. 6)	34 500 (Fig. 7)	Nitrogen	3	600
E5-45C	32 500 (Fig. 6)	36 000 (Fig. 8)	Air	3	600
Average	32 500	35 200			
	Condition ½ H	**Condition ½ HT**			
A5-45	34 800 (Fig. 9)	38 500 (Fig. 10)	Nitrogen	2	600
C4-45	34 000 (Fig. 9)	39 000 (Fig. 10)	Nitrogen	2	600
E3-45	35 000 (Fig. 9)	35 000[a] (Fig. 10)	Nitrogen	2	600
E6-45C	38 500 (Fig. 9)	44 000 (Fig. 11)	Air	2	600
Average	35 600	40 100			
	Condition H	**Condition HT**			
A6-45	37 000 (Fig. 13)	41 800 (Fig. 14)	Nitrogen	2	600
C5-45	35 000 (Fig. 13)	41 000 (Fig. 14)	Nitrogen	2	600
E4-45	36 000 (Fig. 13)	38 900 (Fig. 14)	Nitrogen	2	600
E7-45C	39 000 (Fig. 13)	41 000 (Fig. 15)	Air	2	600
Average	36 800	40 700			

[a] Not included in average because of pitted surface of hardened specimens.

Fatigue Properties:

The fatigue properties of the various materials after precipitation-hardening in accordance with the standard acceptance test were first investigated. For this phase of the work, the test specimens were heat-treated either in air in a muffle furnace or in purified nitrogen in a tubular furnace. All specimens were hardened at 600 F., the condition A material for 3 hr., the rolled tempers for 2 hr. The specimens hardened in purified nitrogen were not tarnished; those in the muffle furnace were oxidized but, in the first series of experiments, this film was not removed prior to test. The fatigue characteristics of the twelve lots of beryllium-copper so heat-treated are summarized in Table III and in the *S-N* diagrams referred to therein.

From the data in Table III it can be seen that the fatigue strength values for material heat-treated in air fall in the same range as those for material heat-treated in purified nitrogen. Hence the two methods may be considered to be essentially equivalent. In these tests, the fatigue strength increased from an average of 32,500 psi. for condition A material to 36,800 psi. for condition H material, an increase of approximately 15 per cent due to cold rolling.[9] Likewise, an increase of about 10 per cent in the fatigue strength of the various tempers was obtained as a result of heat treatment.

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[9] It cannot be said that the condition A material was completely free from any cold-working because microhardness tests made across the 0.032-in. section indicated greater hardness near the surface than at the center. This is probably due to the use of a roll-straightening operation following the solution heat treatment. The effect and magnitude of this cold-working are unknown but are believed to be slight.

In general, there was little variation in the fatigue strength of material of the same temper, regardless of source or tensile strength after hardening. The low fatigue strength of alloy E3-45 after heat treatment was due to bad surface pitting of the material. These pits undoubtedly acted as stress raisers in the hardened material and led to premature failure of some of the specimens. This premature failure was absent in the unaged material where the greater ductility precluded the building up of highly localized stresses. The high fatigue strength of the replacing sample, E6-45C, appears to be associated with a very fine grain size.

When a comparison is made between the fatigue and tensile strength values of the material aged in accordance with the standard acceptance test, some interesting relationships are revealed. In general, the ratio between the fatigue and tensile strength values for a given temper is constant, irrespective of source or variation in composition. This ratio is approximately 0.45 for condition A material, 0.38 for condition ½H material, and 0.33 for condition H material. After heat treatment, the ratio is approximately 0.21 for all three tempers. In this, heat-treated beryllium-copper is similar to the steels where, regardless of condition, the ratio of fatigue to tensile strength is a fairly constant, although somewhat greater, value than that for the beryllium-copper.

TABLE IV.—EFFECT OF AGING TEMPERATURE ON FATIGUE STRENGTH.

Designation	Condition	Fatigue Strength, psi.		
		Unaged	Aged at 600 F.	Aged at 575 F.
C3-45	A	32 500 (Fig. 6)	34 800 (Fig. 7)	31 700 (Fig. 7
C4-45	½H	34 000 (Fig. 9)	39 000 (Fig. 10)	33 700 (Fig. 10
C5-45	H	35 000 (Fig. 13)	41 000 (Fig. 14)	38 000 (Fig. 14
Average Ratio $\frac{\text{Fatigue strength}}{\text{Tensile strength}}$			0.22	0.20

TABLE V.—FATIGUE TEST DATA.

Designation	Condition	Precipitation-Hardening Treatment			Fatigue Strength, psi.	Improvement in Fatigue Strength Resulting from This Treatment as Compared to Acceptance Test, per cent
		Medium	Temperature, deg. Fahr.	Time		
A5-45-II	½HT	Nitrogen	600	2 hr.	38 500 (Fig. 10)	Acceptance test
A5-45-IX	½HT	Salt bath	600	1 hr.	38 500 (Fig. 12)	0
A5-45-X	½HT	Salt bath	780	8 min.	40 500 (Fig. 12)	5
A5-45-XI	½HT	Oil bath	525	45 min.	39 200 (Fig. 16)	2
A6-45-II	HT	Nitrogen	600	2 hr.	41 800 (Fig. 14)	Acceptance test
A6-45-XVIII	HT	Salt bath	660	20 min.	43 000 (Fig. 16)	3
A6-45-XIX	HT	Salt bath	700	20 min.	48 000 (Fig. 16)	15

Heat Treatment at 575 F.:

Since the hardening tests on material obtained from supplier C indicated that peak hardness was obtained at 575 rather than at 600 F., the physical properties of these three alloys also were determined after precipitation-hardening at the lower temperature. In general, the tensile strength was improved by the low-temperature aging treatment, but the fatigue properties were impaired as shown by the data in Table IV. In fact, conditions A and ½H material aged at 575 F. had no better fatigue properties after aging than did the unaged material.

Heat Treatment in Salt and Oil Baths:

Inasmuch as many commercial beryllium-copper springs are precipitation-hardened in salt or oil baths, some tests were made on material heat-treated in this way. These tests were limited to the rolled tempers obtained from supplier A. Because of a shortage of material, it was necessary to make some of the tests on condition ½H material, others on condition HT material. The results of all the physical tests are given in Table II. To facilitate discussion, the fatigue test data are also given in Table V.

TABLE VI. EFFECT OF SURFACE TREATMENT ON FATIGUE STRENGTH.

Designation	Condition	Precipitation-Hardening Treatment		Fatigue Strength, psi.		Improvement in Fatigue Strength, per cent
		Temperature, deg. Fahr.	Time, hr.	No Surface Treatment[a]	After Surface Treatment Indicated	
A4-45	AT	600	3	35 500 (Fig. 7)	43 000 (Polished) (Fig. 8)	21
C3-45	AT	600	3	34 800 (Fig. 7)	47 000 (Polished) (Fig 8)	35
E2-45	AT	600	3	34 500 (Fig. 7)	40 400 (Polished) (Fig. 8)	17
Average				34 900	43 500 (Polished)	24
A5-45	½HT	600	2	38 500 (Fig. 10)	42 500 (Polished) (Fig. 11)	10
C4-45	½HT	600	2	39 000 (Fig. 10)	44 000 (Polished) (Fig. 11)	13
E3-45	½HT	600	2	35 000 (Fig. 10)	41 000 (Polished) (Fig. 11)	17
Average				37 500	42 500 (Polished)	13
A5-45	½HT	600	2	38 500 (Fig. 10)	40 500 (Pickled) (Fig. 12)	5
A5-45	½HT	600	2	38 500 (Fig. 10)	39 700 (Buffed) (Fig. 12)	3
A6-45	HT	600	2	41 800 (Fig. 14)	47 500 (Polished) (Fig. 15)	13
C5-45	HT	600	2	41 000 (Fig. 14)	51 000 (Polished) (Fig. 15)	27
E4-45	HT	600	2	38 900 (Fig. 14)	45 000 (Polished) (Fig. 15)	16
Average				40 200	47 800 (Polished)	19

[a] The control specimens were heat-treated in purified nitrogen, the others in air.

From these tests, it can be seen that material heat-treated in a salt bath for 1 hr. at 600 F. had the same fatigue strength as material heat-treated in accordance with the acceptance test. Heat treatment in salt baths at higher temperatures for shorter periods of time resulted in some increase in fatigue strength with no significant decrease in the tensile properties except for material hardened at 780 F. There was a marked decrease in tensile strength of material heat-treated at this temperature, indicating that the material was overaged. However, there was no loss in fatigue strength and only a slight decrease in yield strength. The high elongation, along with the good elastic and fatigue properties of material heat-treated at 780 F., suggests the desirability of using such high-temperature aging treatments more extensively where adjustment after assembly is required.

Effect of Surface Treatments:

As previously noted, the specimens hardened in air in a muffle furnace were badly oxidized. Since pickling or acid-dipping is the accepted commercial method for removing such films, specimens of alloy A5-45 were so treated after precipitation-hardening for 2 hr. at 600 F. The specimens were cleaned in an alkaline cyanide solution followed by a bright-acid dip.[10] Such pickled specimens had improved fatigue strength as can be seen from the data in Table VI. These data also show the effect on the fatigue strength of buffing and polishing. A cloth wheel and jeweler's rouge were used in the buffing operation, No. 400 Aloxite paper for the polishing.

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[10] In pickling, the specimens were first boiled in a solution containing 10 parts sodium cyanide and 10 parts sodium hydroxide per 100 parts of water, rinsed in hot water, and bright acid-dipped in a solution containing 64 parts water, 64 parts sulfuric acid, 32 parts nitric acid, and 1 part hydrochloric acid, after which they were again rinsed in hot water and dried.

From these data, it can be seen that both pickling and buffing improved the fatigue strength slightly. Polishing with No. 400 Aloxite paper, an operation which involved some work-hardening along with the removal of the oxide film and some surface material, increased the fatigue strength from 13 to 24 per cent. Except for material aged at 700 F., these values reflect the highest fatigue strengths obtained in this investigation and approach the inherent fatigue properties reported by Anderson and Smith (5). While these values are of considerable interest, it is not commercially feasible to use such polishing operations in the manufacture of most beryllium-copper springs. Instead, pickling will probably be used in most cases to remove the oxide film and this operation may be considered beneficial on the basis of these tests.

CONCLUSIONS

1. As can be seen from these studies, there is a marked variation in beryllium content among the ternary beryllium-copper alloys made to the requirements of Tentative Specifications B 120 - 41 T.[3]

2. Despite the variation in beryllium content, all twelve of the commercial ternary alloys studied in the 1945 investigation, including alloys with as little as 1.80 per cent beryllium, could be precipitation-hardened to meet the specified tensile requirements.

3. Alloys with low beryllium content were found to be more sensitive, with respect to time and temperature used for precipitation-hardening, than alloys of higher beryllium content, although satisfactory physical properties were obtained in all cases.

4. Alloys of high beryllium content are more apt to contain beta constituent in the "as received" condition than are alloys of low beryllium content.

5. Despite the variation in beryllium content, the use of different additive elements, and the presence or absence of beta constituent in the "as received" condition, the same fatigue strength values were obtained in the 1945 studies for all materials of the same temper after similar heat treatments. However, there was such a divergence in the values of the other physical properties as to make the interchangeability of the various commercial alloys in engineering applications, based upon average properties, questionable. Except where designs are based upon the minimum physical properties of the poorest alloy, interchangeability can be achieved with the present alloys only by segregating material from each supplier and by using different precipitation-hardening treatments.

6. For material "as received" from the supplier, the ratio between the fatigue and tensile strength was found to be approximately 0.45 for condition A, 0.38 for condition $\frac{1}{2}$H, and 0.33 for condition H.

7. For material hardened at 600 F. in accordance with the acceptance test in Tentative Specifications B 194 - 45 T[4], the ratio between fatigue and tensile strength was found to be approximately 0.21, irrespective of the amount of cold rolling previously done. This ratio differs slightly for the other special heat treatments studied.

8. The fatigue strength of unhardened beryllium-copper strip was increased about 10 per cent as a result of cold rolling to condition H.

9. The fatigue strength of all tempers of beryllium-copper strip was increased approximately 15 per cent as a result of heat treatment at 600 F.

10. While there was little difference in the other physical properties between the alloys tested in 1945 and those tested in 1936, there was a marked improvement in the fatigue strength of the alloys of more recent manufacture.

11. The fatigue strength and the other

physical properties of alloys containing the maximum permissible beryllium content were not impaired by nickel additions up to 2 per cent. However, when the beryllium content was decreased from 2.20 to 2.00 per cent, it was impossible to obtain the specified physical properties in the high-nickel alloys with the recommended heat treatment. In general, the fatigue properties of the high-nickel beryllium-copper alloys were better than those of corresponding tempers of the standard ternary alloys.

12. While overaging had little or no adverse effect on the fatigue properties of beryllium-copper strip, underaging, in most cases, was deleterious. Both overaging and underaging had a marked, but variable, effect on the other physical properties.

13. The fatigue properties were improved by surface treatments subsequent to precipitation hardening. Pickling and buffing resulted in a slight increase in fatigue strength, polishing with No. 400 Aloxite paper in a marked increase in fatigue strength.

14. Short-time, high-temperature heat treatments resulted in better fatigue properties than did standard low-temperature heat treatments. Since such aging treatments are critical with respect to time, they are best applied to individual parts rather than to batch lots of beryllium-copper springs.

Acknowledgments:

The authors wish to thank the various members of the Chemical Laboratories who aided them in this investigation. E. S. Greiner carried out most of the precipitation-hardening treatments, F. G. Foster and R. M. Sample made the metallographic studies, while G. J. Herbert and Miss L. E. Menges supplied the data on the tensile properties.

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DISCUSSION

Mr. H. G. Williams[1] *(written discussion)*.—Messrs. Gohn and Arnold are to be congratulated for their fine contribution to the knowledge of beryllium copper. The evaluation of any alloy requires the painstaking collection of many data. This is particularly true of endurance studies, and the authors and their associates have performed an outstanding service to everyone interested in beryllium copper.

Several points appear to be definitely established by this study:

1. Chemical composition of the alloy does not vitally affect the endurance life as long as sufficient beryllium is available for full hardening.

2. Cold work prior to hardening improves the endurance of beryllium copper, although not as markedly as might be expected.

3. Polishing and "pickling" after hardening increase the endurance limit. The fact that chemical cleaning, which can readily be applied commercially, improves the endurance life is of considerable practical interest.

4. The outstanding fact to be noted is the effect of heat treatment on the endurance life. This is of particular interest to me, since determining the proper heat treatment of beryllium copper for optimum spring performance is my main occupation.

There can be little quarrel in general with the authors' conclusions that underaging is harmful, and that overaging has less damaging effects. The study does not definitely establish the optimum hardening heat treatment for best endurance life, which is not surprising since evidently the authors did not set out to do this. There are, however, several clues shown in Fig. 1 which can be followed up:

1. Reducing the temperature of heat treatment on specimens C3, C4, and C5 to 575 F. lowered the endurance life over the 600 F. test, although the other physical properties were generally improved by the lower temperature heat treatment. In the test (Fig. 1), specimen C3-45 was slightly overaged by the 3-hr. heat treatment at both temperatures, peak hardness coming at about $2\frac{1}{2}$ hr. for 600 F. and $2\frac{1}{4}$ hr. for 575 F.

Material C4-45 was overaged at 600 F. and underaged at 575 F. when heat treated for 2 hr., the peak hardness time being respectively 1 and 3 hr.

Material C5-45 was overaged at both temperatures when heat treated for 2 hr., the peak hardness time being about $\frac{1}{2}$ hr. for 600 F. and $1\frac{1}{2}$ hr. for 575 F.

2. Increasing the temperature of heat treatment on material A6-45 showed a marked increase in endurance life, even though the higher heat-treating temperature slightly lowered the other physical properties. The higher temperature specimens were hardened in a salt bath, so the hardness-response curves of Fig. 1 do not apply, due to the different rate of heating in the salt bath. Mr. Gohn kindly furnished duplicate samples, and the hardness-response curves for salt-bath heating were determined in the Instrument Specialties Co. Laboratory. These tests show peak hardness in salt bath to come in 20 min. at 660 F., 15 min. at 700 F. for sample A6-45. Figures 1 and 22 and

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[1] Chief Metallurgist, Instrument Specialties Co., Inc., Little Falls, N. J.

Table II show the acceptance specimen A6-45-II to be heat treated at peak by 2 hr. at 600 F. in nitrogen. In this group, heat-treated close to peak hardness, the best endurance is found at the highest temperature, the value of 48,000 psi. for the 700 F. heat treatment being the highest found for any unpolished specimen.

3. There is a possibility that the improved endurance at higher temperatures is due to different surface condition from the salt-bath heat treatment. This, however, is not indicated in the test run on specimens A5-45-II and A5-45-IX where the specimens were heat-treated in nitrogen and salt bath of the same temperature but different times, both times corresponding closely to peak-hardness time for each heating medium, with the resulting endurance life reported as identical.

4. Specimen A5-45-X, heat treated at 780 F. for 8 min., was evidently considerably overaged, yet in spite of lower physical properties, it developed slightly better endurance than the acceptance specimens heat treated to peak hardness at 600 F.

This evidence points very definitely to the use of temperatures around 700 F. or higher for best endurance life. The optimum heat-treatment time is probably the same as for peak hardness, but overaging does not cause any rapid drop-off in endurance and may be used where desired for other purposes such as increased ductility or conductivity without excessive sacrifice of endurance life. This is borne out by data collected on a helical coil spring designed for a limited life. This compression spring was tested through a cycle of +3000 to +68,000 psi. torsion stress. These springs were heat treated at 660 F. for varying times. The heat-treatment time, tensile strength, and endurance life follow:

Heat-Treatment Time, min.	Tensile Strength, psi.		Average Spring Life, cycles
10	215 000		454 000
20	217 000	peak	938 000
30	213 000		464 000
80	203 000		615 000
160	195 500		548 000

The evidence of this experiment as shown in the accompanying Fig. 23 points very definitely to peak hardness heat treatment for best endurance life. The loss in endurance life due to overaging is not materially affected by the amount of overaging after the initial drop, even though the overaging is carried to at least eight times the peak heat-treating time. Since the other spring properties of beryllium copper are materially affected by the heat-treating schedule used, it is not surprising to find that endurance follows the same pattern.

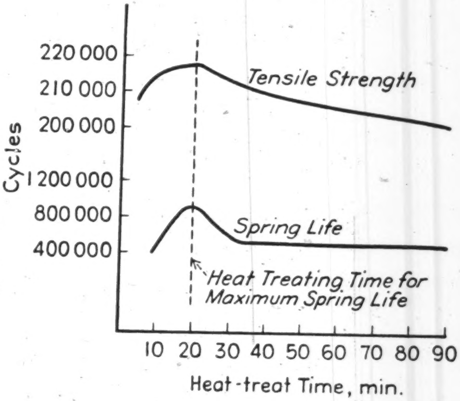

FIG. 23.—Heat-Treatment Time *versus* Endurance Life.

All of these data merely re-emphasize the necessity of heat treating beryllium copper with the guidance of heat-treatment response curves, and the necessity of tailoring the heat treatment to the particular material being used and with regard to the end use of the part being heat-treated.

Referring to the authors' data from their 1942 investigation of the effect of increasing the nickel content of the alloy on the endurance life, there seems to be no assurance that the samples were heat-treated to give best endurance life, and it might be that further study would find considerable improvement from the higher nickel addition.

I should also like to comment on the microstructure shown by materials C3, C4, and C5. There is evidence of a grain-boundary phase which past experience has found to be harmful to spring quality in applications involving spring stability. Such a defect should make the material rejectable. In justice to the supplier, it should be noted that this condition is not typical of material from supplier "C," but is occasionally found in material from all suppliers. It is unfortunate that the minimum physical properties allowed by the A.S.T.M. specification are low enough to allow material with this defect to pass the acceptance test. It is interesting that this structural defect is apparently not as harmful to endurance life as it is to other spring properties.

I feel that the outstanding point developed in this study is the large effect of heat treatment on endurance life and I hope that Messrs. Gohn and Arnold will continue their studies in this direction.

MR. H. S. FREYNIK[2] (*by letter*).—This paper is one of the most important that has been published on beryllium copper. The authors are to be congratulated on such an outstanding contribution to this important subject.

As manufacturers of this alloy, we are particularly interested in two of the conclusions reached. One of these is the marked improvement in the fatigue strength of alloys of recent manufacture. The other concerns interchangeability of the alloys from the various sources, which can only be accomplished by using different precipitation-hardening treatments.

Regarding the first conclusion, it would appear that the precipitation-hardening treatment of 525 F. used in the earlier investigations would explain the lower fatigue values due to underaging. The 1941 investigation does not support this theory since the same hardening treatment was used as in the present investigation. Further complications appear when it is found that the fatigue strength of untreated samples is higher in the recent project.

The presence of ternary additions such as nickel or cobalt, which are now standard practice for the industry, do not explain the recent improvements in fatigue strength, after reference to the various investigations. The 1942 project showed a fatigue value of 43,500 psi. on a binary alloy, which is considerably higher than that reported for binary or ternary alloys in 1936 and 1941. Could there be a possibility that specimens B1-36 and E1-42 were taken from the same experimental lot of material?

Of great significance is the presence of higher impurities in alloys of recent manufacture. This does not imply that higher fatigue values are due to impurities, but rather that the higher impurities did not prove to be deleterious.

One important factor which has not been given the consideration it deserves is the grain size. The recent project reports a grain size for the various samples but past investigations do not. Under these circumstances, an important basis for comparison is not available.

The solution treatment of beryllium copper in the producer's plant has probably been accorded more consideration in the last decade than any other single item of production. The technique usually employed is to obtain a fine grain size together with maximum response to hardening after the precipitation treatment. In the early history of beryllium-copper manufacture, much of the alloy was overheated in the solution treatment and a large grain size obtained. The deleterious feature of this large grain size is that a liquidus segregation is produced at the grain boundaries, which is brittle and will affect the working of the material. Unfortunately, overheated material responds unusually well to precipitation hardening and its possible bad effects are overlooked. Although work from the Bell Laboratories and other sources has conclusively shown improvement in fatigue strength with smaller grain size, the effect upon beryllium copper should reveal some interesting data. We should particularly recommend the inclusion of overheated, large grain size specimens in the investigation.

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[2] Chief Metallurgist, The Riverside Metal Co., Riverside, N. J.

The other conclusion which is of considerable importance concerns the inability of the user of beryllium copper to interchange the alloys from the various suppliers, since different precipitation-hardening treatments are necessary. In our opinion, this is due to the wide variation in additive elements. There is a definite relationship between the quantity of nickel or cobalt present and the peak-hardening temperature of the alloy. An increase in either of these elements increases the temperature at which softening begins. From our experience, we find that the best commercial degree of uniformity is reached when nickel or cobalt is between 0.20 and 0.50 per cent. Correspondingly, the total additive elements of nickel plus cobalt plus iron should be 0.25 to 0.60 per cent.

Our basic criticism of this paper is concerned with the precipitation-hardening treatments. Most of the work was done in a muffle furnace, with and without an artificial atmosphere, and some in a molten salt bath. Commercially, the bulk of this work is done in forced convection furnaces of the recirculating type, without artificial atmospheres, and some in molten salt baths. We are concerned with the possible nonuniformity and localized overheating of muffle furnaces without forced convection.

To emphasize this point further, we should call to your attention the wide difference in tensile strength for material treated at the same time and temperature, in different mediums, from the same original sample. Specimen E4-45-II shows a tensile strength of 188,900 psi. and E4-45-XIII a value of 145,500 psi. after a treatment at 600 F. for 2 hr. in nitrogen and muffle furnaces, respectively. Correspondingly, specimens C3-45-I and C3-45-XII show values of 154,200 and 132,400 psi., respectively. This wide variation gives us much concern, and we wonder whether the cause is associated with nonuniformity of material or the precipitation-hardening treatment.

With reference to testing procedure, we find marked discrepancies between characteristics of the various alloys as illustrated in Fig. 1 and the physical properties reported in Table II. In Fig. 1, alloys C3-45-A and C5-45-H fail to meet required minimum values when treated at 600 F. In Table II, these same alloys treated at the same temperature do meet the required minimums. We wonder how this can be explained.

MR. G. R. GOHN (*author's closure*).—In general, the authors concur with Mr. Williams' conclusion that, within the present specification limits, variations in composition do not markedly affect the fatigue life of heat-treated beryllium-copper strip. However, such variations in composition, particularly a deficiency in the amount of additive elements, may materially affect the other physical properties. As stated by Mr. Williams, the authors were not primarily concerned with the determination of the optimum heat treatment for the various beryllium-copper alloys studied. Instead, we undertook to determine the fatigue characteristics and other physical properties of a number of different lots of beryllium-copper strip when heat-treated as specified in the standard A.S.T.M. acceptance test at 600 F. and when heat-treated in accordance with certain commercial practices at other temperatures.

While the authors' data show superior fatigue properties for material heat-treated at 700 F., it should be pointed out that heat treatment at this temperature or for peak hardness may develop other less desirable properties—for example, there may be a marked loss in ductility or even embrittlement of the material. In the authors' opinion, heat treatment at the lower temperatures specified in the A.S.T.M. acceptance test will permit the hardening of all material, regardless of source, to the specified physical properties provided, of course, that the composition is within the A.S.T.M. limits, and will produce parts having the best over-all physical properties for general use. For specific applications where maximum hardness and fatigue strength are desired, deviations from this procedure may be justified.

While our data show that, by heat treatment at 700 F., it is possible to increase the fatigue strength of beryllium-copper by as much as 30 per cent over that of the unheat-treated material, it should be noted that the fatigue strength was increased 13 per cent by cold rolling alone for condition H material whereas the gain by conventional heat treatment was only 15 per cent. Only by combining cold working and heat treatment are the high fatigue properties, noted in these studies, obtained.

Mr. Freynik states that the data obtained in the 1941 investigation fail to support the authors' theory that underaging (heat treatment at 525 F. for only 1 or 2 hr.) was responsible for the poor fatigue characteristics observed in the earlier (1936) studies. To justify our conclusion, we should like to point out that, while the improved fatigue characteristics observed in the 1945 studies as compared to those reported by Greenall and Gohn[3] could be due either to differences in heat treatment or to improvement in materials of more recent manufacture, this cannot be said of the improvement noted in the 1945 studies as compared to the 1941 studies. In both the 1941 and the 1945 studies, the heat treatments were the same, namely, 600 F. for 3 hr. for condition A material and 2 hr. for the rolled tempers. It is true that the 1941 studies dealt with materials heat treated in a salt bath, while those made in 1945 were generally concerned with materials heat treated in air or nitrogen atmospheres. However, since the heat-treatment times given in Table II represent time at heat, the use of these different media should not, in our opinion, have any significant effect. It must therefore be concluded that the better fatigue properties noted in the 1945 tests over those reported in 1941 were due primarily to improvement in materials.

This conclusion is further justified by reference to the data on the unheat-treated materials studied in 1936 and those studied in 1945. Condition ½H material of earlier manufacture had an average fatigue strength of 23,100 psi., whereas material of recent manufacture had a fatigue strength of 35,600 psi. Similarly, condition H material studied in 1936 had a fatigue strength of 27,000 psi., whereas material of recent manufacture had a fatigue strength of 36,800 psi. Accordingly, we believe that we are justified in making the statement that "there was a marked improvement in the fatigue strength of the alloys of more recent manufacture."

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[3] See reference (4) of the paper.

Mr. Freynik asks whether specimens designated B1-36 and E1-42 were taken from the same lot of material. Specimens B1-36 were taken from an experimental lot of beryllium-copper made by the Bell Telephone Laboratories prior to 1936 and none of this material was available at the time the 1942 studies were made. Specimens designated E1-42 were made by an outside supplier under carefully controlled conditions, to produce a binary material having the best possible fatigue properties and was intended as a control lot for the studies on the effect of nickel on the physical properties of beryllium-copper. No other commercial binary alloy of comparable physical properties has been studied.

The effect of a deficiency of nickel or cobalt is illustrated by the poor response of alloys C3, C4, and C5 to heat treatment (see Table II and Fig. 1) and by the large grain size shown in the micrographs for specimens C3-45, C4-45, and C5-45 (Figs. 17, 18, and 19). We believe that Mr. Freynik is correct when he suggests that 0.20 to 0.50 per cent of additive elements (nickel or cobalt) should be present for uniform response of the various alloys to the standard heat treatment and that, if iron is included as an additive element, both the minimum and maximum percentages should be raised.

We concur with Mr. Freynik that the effect of grain size on the fatigue characteristics of non-ferrous alloys is marked and not very well understood. Some data on this subject have been presented by Greenall and Gohn[3] but further work on the effect of grain size on fatigue is definitely needed.

Mr. Freynik has expressed concern over the effect on the physical properties of possible nonuniformity and localized over heating in the muffle furnace used in our heat treatments in the absence of forced convection. Data are cited by him on specimens E4-45-II, E4-45-XIII, C3-45-I, and C3-45-XII to suggest that the variations in physical properties reported by the authors was due to nonuniformity of the heat treatment rather than nonuniformity of the materials studied. Therefore we have reanalyzed the tension test data for the beryllium-copper alloys heat-treated at 600 F. in the 1945 studies, to determine whether there are differences in physical properties which can be attributed to nonuniformity in heat treatment rather than nonuniformity in material. These data are shown in Table VII.

The specimens listed in Table VII as being heat-treated in a nitrogen atmosphere were clamped in a jig, placed in a quartz tube which was closed at one end and mounted within a tubular furnace having a uniform heating zone over a length of at least 12 in. Purified nitrogen was introduced into the closed end of the quartz tube and a small flow of nitrogen maintained during heat treatment. The temperature of the furnace was regulated by an automatic controller recorder while the temperature of the specimens was measured with a thermocouple buried in the charge. With this setup the variation in temperature during heat treatment did not exceed 2 to 3 C. (3 to 5 F.). Hence no localized heating or overheating was possible in the case of heat treatments I or II. Each lot of material was separately heat-treated.

In the case of heat treatments XII and XIII, which were carried out in a muffle furnace, the specimens were bound together, wrapped in copper foil and placed in a tray supported from the controlling thermocouple near the center of the muffle furnace. All condition A material was heat treated at one time and all of the rolled tempers were heat treated together as a second charge.

The temperature was regulated by an automatic controller recorder with supplementary hand control but, even without the latter, the temperature variation did not exceed ± 10 C. (± 18 F.). With hand control, the variation was considerably less. Heat treatment XV was carried out in a commercial, forced convection type of muffle furnace such as that recommended by Mr. Freynik.

TABLE VII. COMPARISON OF TENSILE PROPERTIES OF BERYLLIUM-COPPER STRIP PRECIPITATION-HARDENED AT 600 F. UNDER VARIOUS CONDITIONS.

(1945 Investigation)

Designation	Precipitation-Hardening Time, hr.	Type of Furnace Used	Medium	Tensile Strength, psi.
		CONDITION AT MATERIAL		
A4-45-I	3	Quartz tube	Nitrogen	181 300
A4-45-XII	3	Muffle	Air	170 500
C3-45-I	3	Quartz tube	Nitrogen	154 200
C3-45-XII	3	Muffle	Air	132 400
E2-45-I	3	Quartz tube	Nitrogen	151 300
E2-45-XII	3	Muffle	Air	144 300
		CONDITION ½HT MATERIAL		
A5-45-II	2	Quartz tube	Nitrogen	200 100
A5-45-XIII	2	Muffle	Air	200 900
C4-45-II	2	Quartz tube	Nitrogen	174 300
C4-45-XIII	2	Muffle	Air	180 400
E3-45-II	2	Quartz tube	Nitrogen	179 800
E3-45-XIII	2	Muffle	Air	163 900
		CONDITION HT MATERIAL		
A6-45-II	2	Quartz tube	Nitrogen	199 300
A6-45-XIII	2	Muffle	Air	196 700
A6-45-XV	2	Forced convection	Air	195 500
A6-45-IX	1	...	Salt Bath	195 800
C5-45-II	2	Quartz tube	Nitrogen	180 500
C5-45-XIII	2	Muffle	Air	186 400
E4-45-II	2	Quartz tube	Nitrogen	188 900
E4-45-XIII	2	Muffle	Air	145 500

Before analyzing the data shown in the accompanying Table VII, the authors plotted the tensile strength Rockwell hardness data given in Table II of the paper and found that a variation of one Rockwell number (C scale) was equivalent to a variation of approximately 5000 psi. in tensile strength. If we now consider the data given in Table VII along with the data shown in Fig. 1 of the paper (which indicate the response of the various alloys to hardening) it can be seen that the low tensile strength values obtained for specimens of condition A material heat-treated in the muffle furnace can be explained on the assumption that the temperature in the muffle furnace was higher than that in the tubular furnace. A difference of less than 18 F. would explain the low tensile strength values observed for heat treatment I.

As previously stated, all samples of the rolled tempers were heat treated simultaneously in the muffle furnace. If the temperature in the muffle furnace was as much as 18 F. lower than the temperature of the quartz tube furnace (600 F.) the differences noted under condition ½HT material for samples designated A5, C4, and E3 can be explained. This assumption does not, however, explain the differences noted under condition HT material for samples designated C5 and E4. In the authors' opinion these differences are due to a nonuniform response of the latter two materials to heat treatment.

The Rockwell hardness values reported in Fig. 1 were obtained on small 1-in. square samples heat-treated in the tubular furnace at a different time than the tension test specimens for which corresponding Rockwell hardness values are given in Table II. In view of the marked effect of small variations in temperature on the physical properties as discussed in the preceding paragraphs we believe that there is no real discrepancy among the hardness data reported in Fig. 1 and those given in Table II.

THE FATIGUE STRENGTH OF SOME MAGNESIUM SHEET ALLOYS*[1]

By L. R. Jackson[2] and H. J. Grover[2]

Synopsis

This paper contains data on the direct-stress fatigue strength, and notch sensitivity of magnesium sheet alloys of the J-h, J-a, M-h, M-a, and FS-h and FS-a types.

One of the projects undertaken by the War Metallurgy Division of the National Defense Research Committee, during the war, was the evaluation of magnesium sheet alloys as possible materials for the construction of aircraft. The portion of this project dealing with the reaction of such materials to repeated stresses was assigned to Battelle Memorial Institute. This paper is concerned with the direct-stress fatigue strength of various magnesium sheet alloys, an evaluation of their notch sensitivity as revealed by repeated stress, and other pertinent information. A subsequent paper deals with the fatigue strength of various types of joints in the alloys investigated.[3]

Materials Investigated

The purpose of the investigation was not to attempt to develop new alloys but to evaluate materials which were already in significant commercial production. On this basis, the alloys listed in Table I were selected. It will be noted in Table I that there was no single uniform designation for the various types of alloys. Accordingly, all the various designations, current at the time, are listed for each alloy. In the body of the paper only one designation is used, and this denotes the source of the material. For example, all tests labeled with Dow Chemical Co. designations were performed on material obtained from that company.

Types of Test Specimens

Figure 1 shows photographs of various types of test specimens that were used in the investigation.

Dimensions connected with the various types of test specimens are listed in Table II. In tests described in the paper, test specimens will be designated by type number. In practically all cases, the tests were performed on 0.064-in. thick sheet. Unless otherwise designated, this thickness is to be understood in all descriptions of tests.

Tensile Properties of Sheet Materials

Table III summarizes average static tensile properties of the various types of sheet materials and test specimens used in the investigation of fatigue properties.

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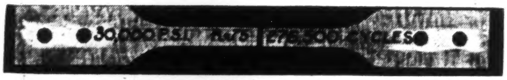
* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

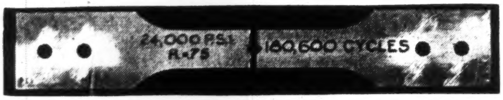
[1] This paper is based on work done for the Office of Scientific Research and Development, under Contract No. OEMsr-729 with Battelle Memorial Institute, Columbus, Ohio.

[2] Supervisor and Assistant Supervisor, respectively, Battelle Memorial Institute, Columbus, Ohio.

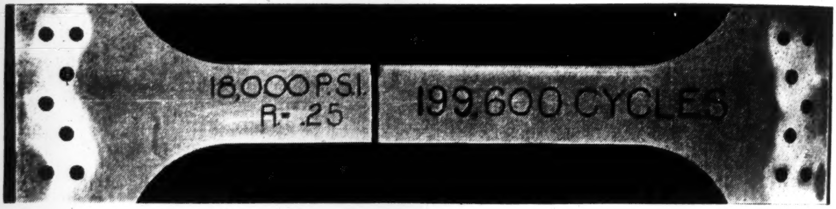
[3] H. J. Grover and L. R. Jackson, "The Fatigue Strength of Lap Joints in Some Magnesium Sheet Alloys," *Preprint No. 28*, Forty-Ninth Annual Meeting, Am. Soc. Testing Mats., June 24–28, 1946.


30,000 P.S.I. R=.75
275,500 CYCLES

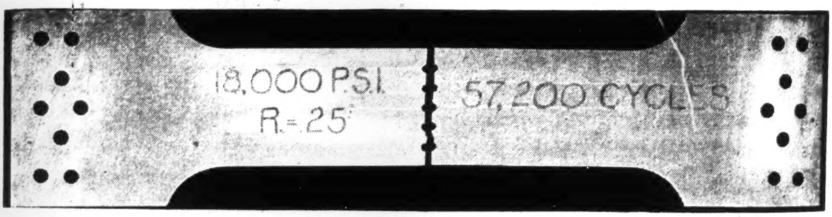
Type 1


24,000 P.S.I.
R=.75
160,600 CYCLES

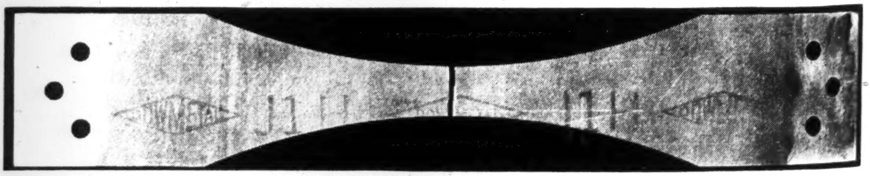
Type 2


18,000 P.S.I.
R=.25
199,600 CYCLES

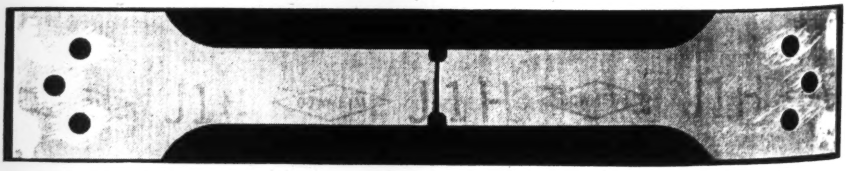
Type 3


18,000 P.S.I.
R=.25
57,200 CYCLES

Type 4



Type 5



Type 6

FIG. 1.—Illustrations of Various Types of Test Sections.

(See Table II for dimensions)

TABLE I.—NOMINAL COMPOSITIONS OF MAGNESIUM SHEET ALLOYS USED IN INVESTIGATION OF FATIGUE PROPERTIES.

Designation of Alloy						Nominal Composition, per cent			Temper
A.S.T.M.[a]	Dow Chemical Co.	A.M.S.	American Magnesium Co.	S.A.E.	Electron	Al	Mn	Zn	
AZ61X(H)	J-1h	4381	57S-h	....	AZM	6.5	0.2	0.7	Hard-rolled
AZ61X(A)	J-1a	4380	57S-o	....	AZM	6.5	0.2	0.7	Annealed
M1(H)	M-h	4370h	3S-h	51	AM503	...	1.5	..	Hard-rolled
M1(A)	M-a	4370a	3S-o	51	AM503	...	1.5	...	Annealed
AZ31X(H)	FS-1h	....	52S-h	52	AZ31	2.8	0.3	1.0	Hard-rolled
AZ31X(A)	FS-1a	....	52S-o	52	AZ31	2.8	0.3	1.0	Annealed

[a] Tentative Specifications for Magnesium-Base Alloy Sheet (B 90 – 45 T), 1945 Supplement to Book of A.S.T.M. Standards, Part I, p. 200.

TABLE II.—DIMENSIONS OF TEST SPECIMENS.

(See Fig. 1 for illustrations)

Type	Over-all Length, in.	Length of Test Sections, in.	Width of Test Sections, in.	Remarks
No. 1	12	6	0.5	
No. 2	12	6	1.0	a
No. 3	21	15	2.00	
No. 4	21	15	3.00	b
No. 5	17		1.00	c
No. 6	17	11	1.541	d

[a] Hole 0.190-in. dameter drilled in center of 1-in. wide sections.

[b] Five holes 0.190-in. diameter spaced 0.5 in.

[c] Sweep radius 12 in.

[d] Width of unnotched section 1.541 in. Edge notches 0.25 in. deep, 0.125-in. radius. Net width at center, 1.041 in.

From Table III it will be noted:

1. The materials investigated show no substantial difference between the static properties parallel and perpendicular to the rolling direction.

2. The static notch sensitivity as revealed by tension tests on sheets with holes does not appear to be abnormally high. There is sufficient ductility that the strength of the sheets is not impaired by holes. This is contrary to results reported by Beck[4] who quoted data on

TABLE III.—STATIC TENSILE PROPERTIES OF MAGNESIUM SHEET ALLOYS.

(All test specimens cut parallel to direction of rolling unless otherwise noted.)

Type of Alloy	Type of Test Specimen	Sheet Thickness, in.	Proportional Limit, psi. (0.01 per cent Offset)	Yield Strength, psi. (0.2 per cent Offset)	Tensile Strength, psi.	Elongation in 2 in., per cent	Remarks
1J-1h	1	0.125	18 000	32 875	45 550	10	b
1J-1h	1	0.064	18 000	32 050	46 250	12	b
1J-1h	3	0.064	18 000	31 525	44 300	13	b
1J-1h	3	0.064	18 000	32 900	44 800	11	a
1J-1h	5	0.064	....	32 060	45 900	13.6	b
2J-1h	5	0.064	....	38 970	48 050	3.2	b
J-1a	1	0.064	12 000	24 225	42 500	20	
J-1a	3	0.064	12 000	23 375	41 000	13	
J-1a	3	0.064	12 000	26 900	42 900	19	a
Mh	1	0.064	10 000	26 700	35 600	7	
Mh	3	0.064	10 000	26 600	35 700	14	
Mh	3	0.064	10 000	23 000	35 850	12	a
Ma	1	0.064	9 500	19 500	33 700	27	
1FS-1h	1	0.064	19 000	37 500	45 800	5	
1FS-1h	5	0.064	....	38 120	46 200	5	
1FS-1a	5	0.064	....	30 130	44 180	12	c
2FS-1h	5	0.064	....	33 950	44 920	10	
AMC52S-H	5	0.064	....	36 500	43 300	11	
AMC52S-O	5	0.064	....	20 300	37 100	21	
1J-1h	2	0.064	....	37 480	42 285	2	
J-1h	4	0.064	....	39 775	45 500	1	
J-1h	4	0.064	....	44 700	49 050	1	a
J-1a	4	0.064	....	31 275	39 250	3	
J-1a	4	0.064	...	34 250	41 300	3.5	a
Mh	4	0.064	....	32 400	38 100	2	
Mh	4	0.064	....	30 575	38 700	2	a

[a] Transverse to rolling direction.

[b] A number preceding the alloy type number indicates the lot of material when more than one lot was used.

[c] The properties of this item suggest that it should be designated as FS-1h — the designation given is the one supplied by the manufacturer.

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[4] E. A. Beck, "The Technology of Magnesium, and Its Alloys," F. A. Hughes and Co., London, England p. 187 (1940).

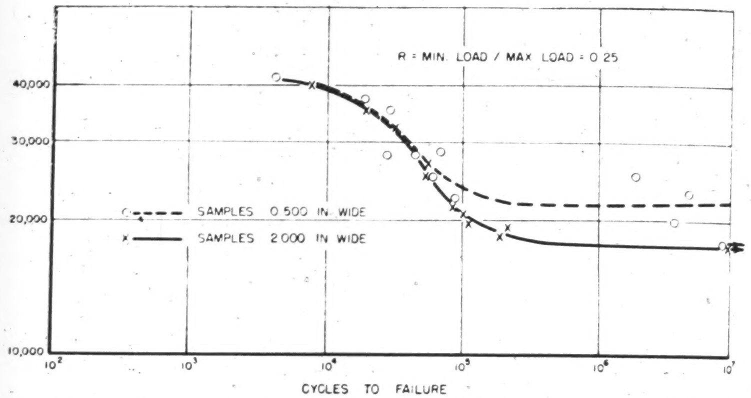

FIG. 2.—Fatigue Curves Showing Specimen "Width Effect" for Specimen of J-1h Cut Parallel to Rolling Direction. (Type 1 and Type 3 Test Specimens.)

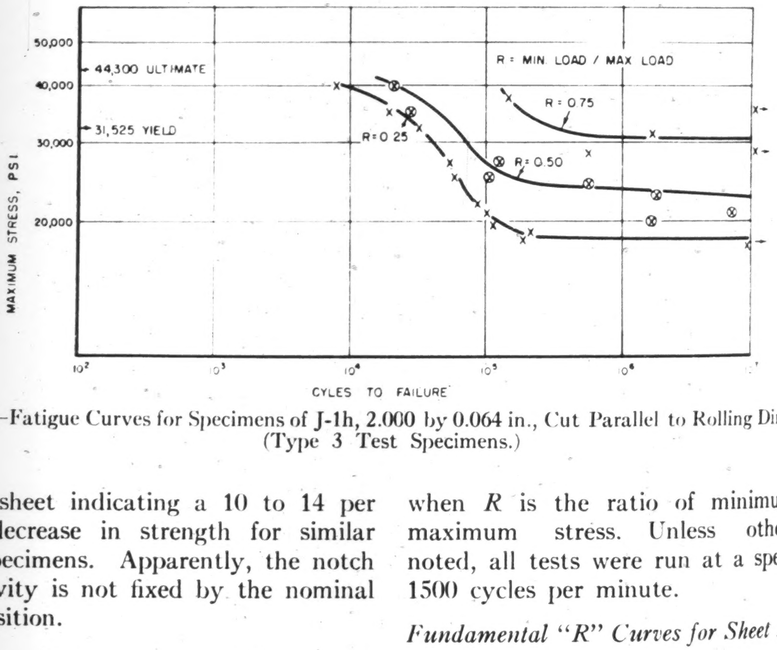

FIG. 3.—Fatigue Curves for Specimens of J-1h, 2.000 by 0.064 in., Cut Parallel to Rolling Direction. (Type 3 Test Specimens.)

AZM sheet indicating a 10 to 14 per cent decrease in strength for similar test specimens. Apparently, the notch sensitivity is not fixed by the nominal composition.

FATIGUE PROPERTIES OF MAGNESIUM SHEET ALLOYS

All fatigue tests reported in this paper were performed on Krouse direct-stress fatigue machines. The tests were performed at a constant ratio of minimum to maximum stress. Curves obtained from such tests are known as *R* curves when *R* is the ratio of minimum to maximum stress. Unless otherwise noted, all tests were run at a speed of 1500 cycles per minute.

Fundamental "R" Curves for Sheet Materials:

A considerable amount of data was obtained on the various sheet materials. These are illustrated in Figs. 2 to 10.

Figure 2 compares the fatigue strength of J-1h alloy using type 1 (0.5 in. wide) and type 3 (2 in. wide) test specimens. The narrow test specimens show higher

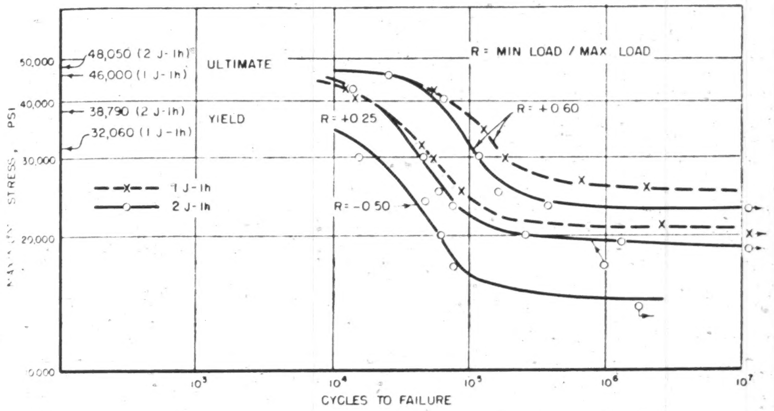

FIG. 4.—Fatigue Curve for Specimens of J-1h Sheet. (Type 5 Test Specimen.)

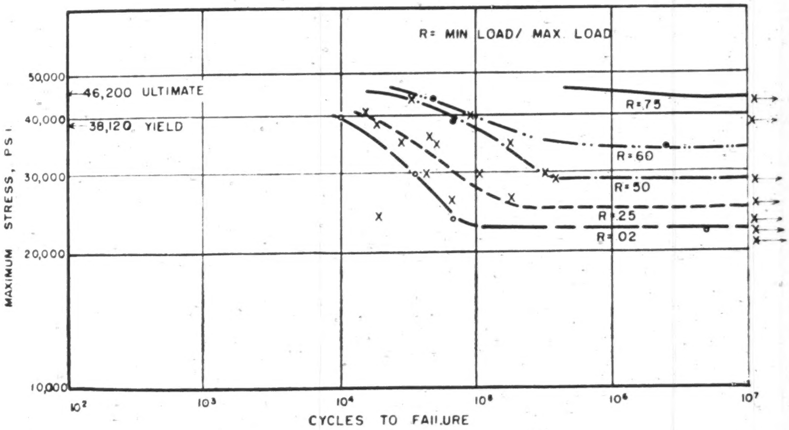

FIG. 5.—Fatigue Curves for Specimens of 1FS-1h. (Type 5 Test Specimens.)
(Specimens 1.000 by 0.064 in. at center section)

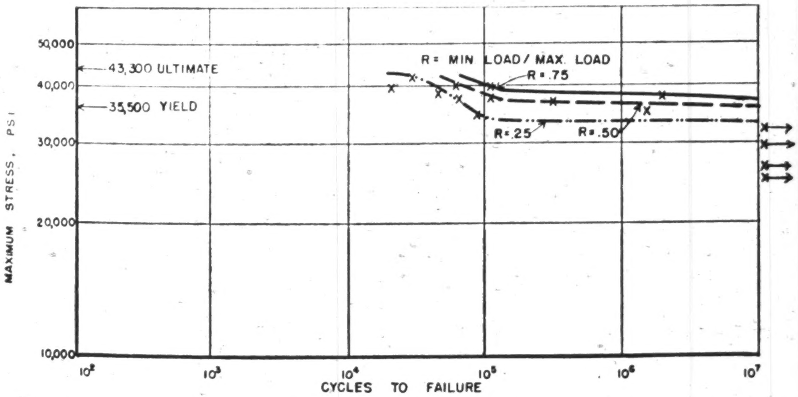

FIG. 6.—Fatigue Curves for Specimens of AMC52S-H. (Type 5 Test Specimens.)
(Specimens 1.000 by 0.064 in. at center section)

strength at long lifetimes, although there is some indication that the results on 2-in. wide test specimens lie at the lower limit of the scatter band for the narrow test specimens.

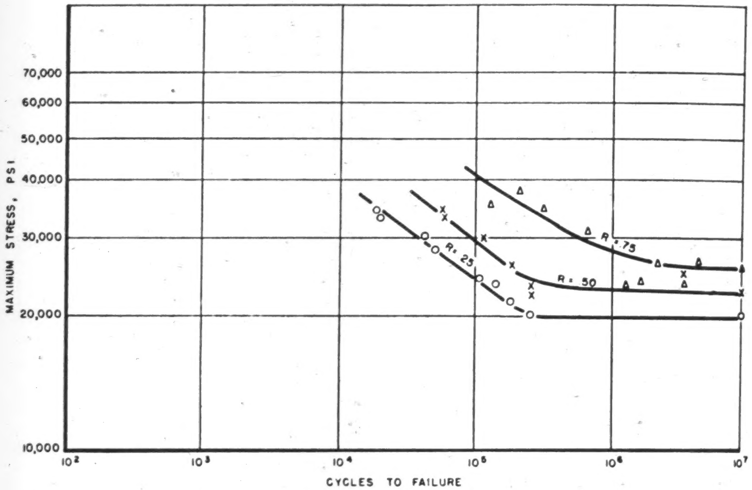

FIG. 7.—Fatigue Curves for Specimens of J-1a. (Type 1 Test Specimens.)
(Specimens 0.5 in. wide, 0.064-in. sheet cut parallel to rolling direction)

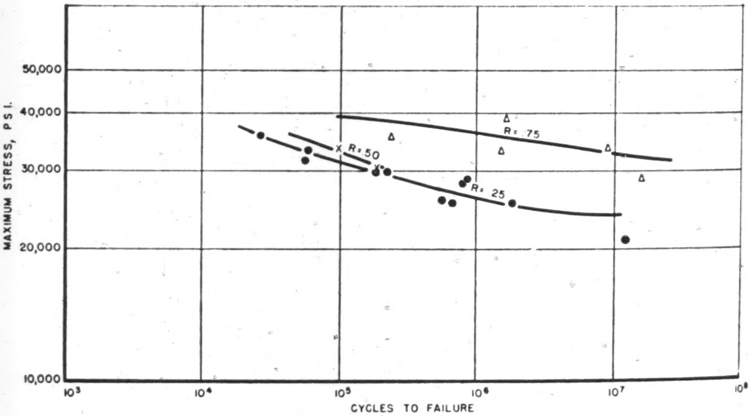

FIG. 8.—Fatigue Curves for Sheet Specimens of M-h Alloy. (Type 1 Test Specimens.)
(Specimens 0.5 in. wide, 0.064 in. sheet cut parallel to rolling direction)

Figure 3 shows more detailed information on type 3 test specimens, and Fig. 4 shows results with type 5 test specimens all with J-1h alloy. Figure 4 compares results with two lots of material. A few check tests with test specimens of 1J-1h alloy cut perpendicular to the direction of rolling indicated that the transverse fatigue properties are not distinguishable from the longitudinal properties.

Figure 5 shows fatigue test results on 1FS-1h alloy; these should be compared with Fig. 6 which shows results with

AMC52S-H alloy. These results were obtained with type 5 test specimens.

Figures 7, 8, and 9 show fatigue test results on alloys J-1a, M-h, and M-a respectively, obtained with type 1 test specimens. Figure 10 compares fatigue test results on 1FS-1a alloy with results on AMC52S-O using type 5 test specimens.

Light metal alloys, such as those of aluminum and magnesium, apparently do not exhibit an "endurance limit" such as is found in steels. However, as can be seen from Figs. 2 to 10, magnesium alloys have a more well-defined "knee" in *S-N* curves than do aluminum alloys and, in this respect, more closely resemble steels.

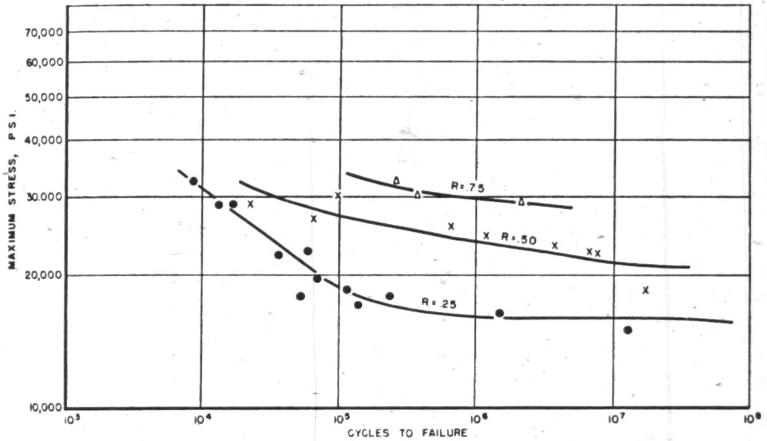

FIG. 9.—Fatigue Curves for Sheet Specimens of M-a Alloy. (Test 1 Test Specimens.)
(Specimens 0.5 in. wide, 0.064-in. sheet cut parallel to rolling direction)

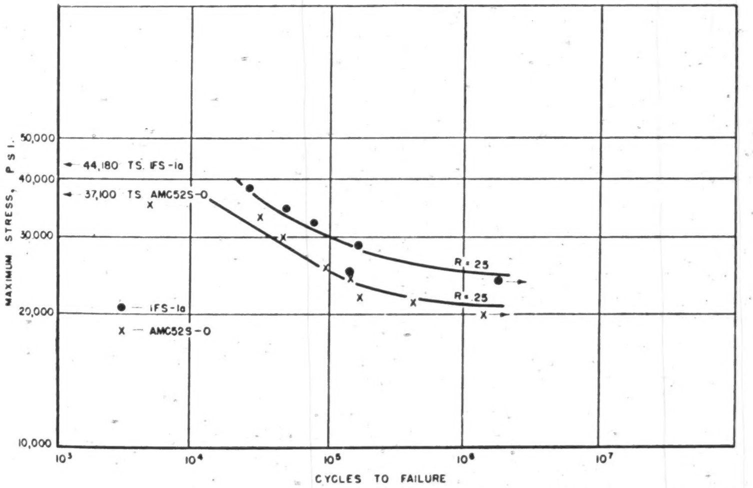

FIG. 10.—Fatigue Curves for Sheet Specimens of 1FS-1a and AMC52S-O Alloys at $R = 0.25$. (Type 5 Test Specimens.)
(Specimens 1 in. wide at center, 0.064 in. thick)

In general, the relation between tensile strength and fatigue strength does not appear to be as well defined as in steels. This is particularly striking when, for example, Mh (Fig. 8) is compared with either of the stronger alloys J-1h (Fig. 2) or 1FS-1h (Fig. 5), and again in comparing 1FS-1h (Fig. 5) with its counterpart AMC52S-H (Fig. 6). It will be noted that the statically stronger of these two alloys has the lower fatigue strength. It also has less ductility. Apparently the ductility (or something that goes along with ductility) of the alloys is a factor in determining the fatigue strength. This is also true in aluminum alloys where fatigue tests indicate that the statically

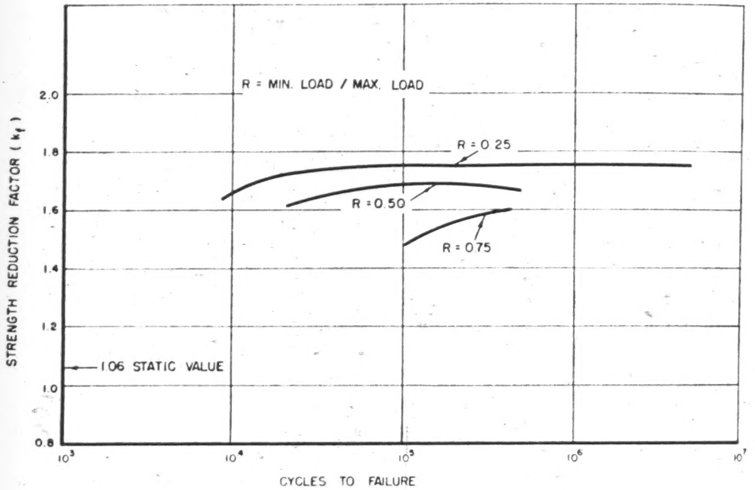

Fig. 11.—Fatigue-Strength Reduction Factors for J-1h Specimens 1.000 by 0.064 in. with One Rivet Hole 0.190 in. in Diameter. (Type 2 Test Specimens.)

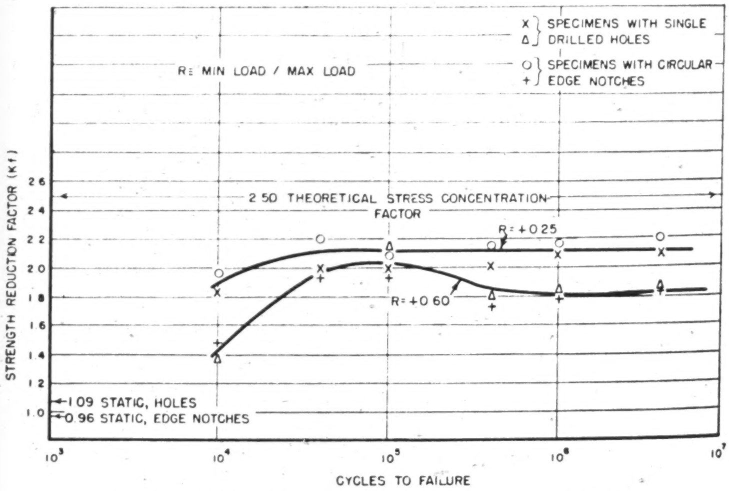

Fig. 12.—Fatigue-Strength Reduction Factors of 1J-1h.

stronger, but less ductile, 75S-T type alloy has no better fatigue strength than 24S-T.

In order to have a more direct comparison with aluminum alloys, a few tests were run on some aluminum sheet alloys using the same shape test specimens and the same testing machines at a stress ratio $R = 0.25$. Comparisons are made in Table IV. For lifetimes in the order of 1,000,000 to 10,000,000 cycles, the magnesium alloys will withstand a greater percentage of their respective static strengths than Alclad 24S-T, although they are relatively not so strong as the bare 24S-T.

TABLE IV.—COMPARISON OF FATIGUE STRENGTH OF MAGNESIUM AND ALUMINUM ALLOYS FOR DIRECT-STRESS FATIGUE CURVES TAKEN AT A RATIO $R = 0.25$.

Alloy	Tensile Strength, psi.	Maximum Stress, psi for $R = 0.25$			Ratio of Fatigue Strength to Tensile Strength		
		10^5 Cycles	10^6 Cycles	10^7 Cycles	10^5 Cycles	10^6 Cycles	10^7 Cycles
1J-1h	45 550	24 000	20 500	20 500	0.53	0.45	0.45
J-1a	42 500	24 000	20 000	20 000	0.56	0.47	0.47
1FS-1h	45 800	27 000	24 000	24 000	0.59	0.52	0.52
1FS-1a	44 180	30 000	25 000	....	0.68	0.56	....
AMC52S-H	43 300	33 000	32 000	32 000	0.76	0.74	0.74
AMC52S-O	37 100	25 000	20 500	20 500	0.67	0.55	0.55
M-h	35 600	31 000	26 000	24 000	0.87	0.73	0.67
M-a	33 700	19 000	16 000	16 000	0.56	0.47	0.47
24S-T Alclad	68 000	50 000	29 000	25 000	0.73	0.42	0.37
24S-T bare	73 700	60 000	52 000	51 000	0.81	0.70	0.69

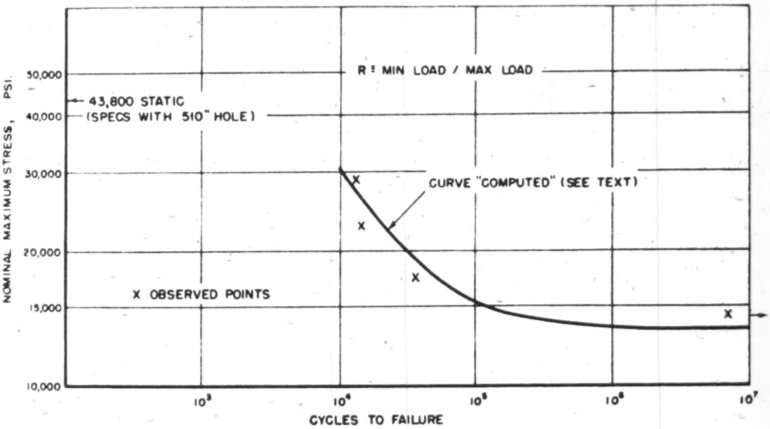

FIG. 13.—Fatigue Curve for 1J-1h Specimens with Large Diameter Holes. (Modified Type 2 Test Specimens.)
(Center 1.000 in. wide, single hole, 0.510-in. diameter)

Notch Sensitivity Under Repeated Stress:

Fatigue tests were run with test specimens containing a single drilled hole (type 2, Fig. 1) and with semicircular notches at the edge of the test specimen (type 6, Fig. 1). These two types of stress raisers were designed to have a geometrical stress-concentration factor of 2.5 as computed by the methods described by Neugebauer.[5]

The geometrical stress-concentration factor for the test specimens containing 5 holes (type 4, Fig. 1) is not known.

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[5] G. H. Neugebauer, "Stress-Concentration Factors in Fatigue and Their Effects on Design," *Product Engineering*, Vol. 14, pp. 82–87 and 168–172 (1943).

In all cases described here, the holes were drilled holes. That is, they were neither reamed nor polished since it was desired to evaluate their effect under conditions approximating service.

The strength-reduction factor (or stress-concentration factor) *Kf* as measured by fatigue tests is defined as the ratio

$$Kf = \frac{\text{Maximum nominal stress unnotched}}{\text{Maximum nominal stress notched}}$$

where both stresses are based on the net section of the test specimen, and are determined at the same *R* value and the same lifetime.

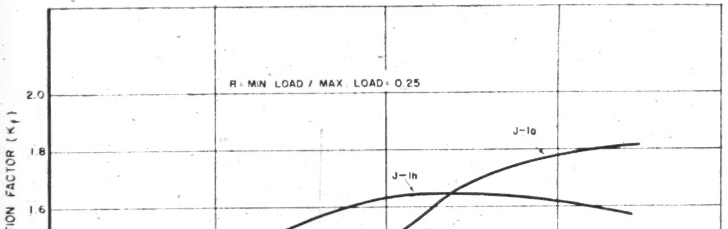

FIG. 14.—Fatigue-Strength Reduction Factors for Specimens 3.000 by 0.064-in. Cut Parallel to Rolling, with 5 Rivet Holes 0.190 in. in Diameter. (Type 4 Test Specimens.)

Figure 11 shows strength-reduction factors at various stress ratios for J-1h alloy (type 2). Figure 12 compares the effect of a single drilled hole (type 2) with semicircular edge notches (type 6). Both types produce very similar effects. Figure 13 compares the effect of another variation in type of stress raiser on J-1h alloy. This additional type was similar to type 2, except that the center hole was of 0.510-in. diameter instead of 0.190 in. and had a theoretical stress-concentration factor of 2.11 instead of 2.50. In Fig. 13 the solid curve was drawn by multiplying values from type 2 test specimens by the ratio $\frac{2.50}{2.11}$, while the points were obtained on actual test specimens having the theoretical stress-concentration factor of 2.11. From Figs. 11, 12, and 13 it may be observed:

1. That the "stress-concentration factor" measured in fatigue is lower for short lifetimes than for long lifetimes and that all values are lower than the geometric stress-concentration factor. This result is consistent with what has been observed on other materials.

2. That results, using different geometrical stress-concentration factors or the same factor obtained in different ways, are consistent with each other.

Figure 14 compares J-1h, J-1a, and M-h alloys using type 4 test specimens cut parallel to the direction of rolling and Fig. 15 makes the same comparison transverse to the rolling direction.

On comparing Fig. 14 with Fig. 11, it will be noted that the spacing between holes in the 5-hole test specimens is apparently such that the stress-concentra-

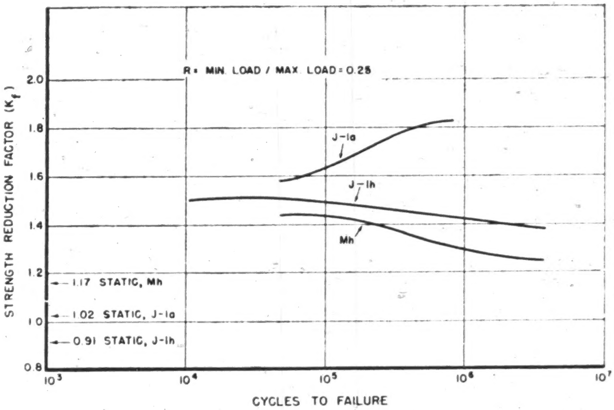

FIG. 15.—Fatigue-Strength Reduction Factors for Specimens 3.000 by 0.064 in. Cut Transverse to Rolling, with 5 Rivet Holes, 0.190 in. in Diameter. (Type 4 Test Specimens.)

tion factor is reduced by mutual interference. Figures 14 and 15 indicate again that the transverse strength is not inferior to the strength in the rolling direction.

At the time these tests were made, not enough material was available of the FS or AMC52S alloys to make a detailed investigation of the notch sensitivity of these alloys. Beck (see footnote 4, p. 218) shows results comparing AZM (similar to J type) with AZ31 (similar to FS or AMC52S type), indicating that the notch sensitivity of the AZ31 type is somewhat lower than for the AZM type.

A few tests were made with aluminum sheet alloys under the same conditions as the tests on the magnesium alloys in order to have a comparison with other constructional materials on the same basis. The comparison is made in Table V. The "notch-sensitivity index" used in Table V is defined as:

$$\text{Sensitivity index} = \frac{\text{Maximum measured stress-concentration factor}}{\text{Theoretical stress-concentration factor}}$$

The "maximum measured stress-concentration factor" is taken from Fig. 11 at a ratio of $R = 0.25$ for the magnesium alloy J-1h and from similar data on the aluminum alloys. This method of defining notch sensitivity was proposed by Beck (see footnote 4, p. 215). While a direct comparison between the aluminum and magnesium alloys was made only with J-1h, by inspection of Figs. 14 and 15 it is seen that none of the magnesium alloys tested appears to be abnormally notch sensitive as compared with the aluminum alloys. The differences in notch-sensitivity index, as shown in Table V, should not be considered to be significant. As Beck (see footnote 4, p. 215) points out, this method of defining notch sensitivity is open to question and it should only be considered capable of revealing gross differences. Within the range of values given in

TABLE V.—COMPARISON OF NOTCH SENSITIVITY OF MAGNESIUM OR ALUMINUM ALLOYS.
(Theoretical stress-concentration factor in all cases = 2.5)

Alloy	Notch-Sensitivity Index
J-1h	0.70
24S-T bare	0.83
24S-T Alclad	0.77

Table V, the materials should be considered to be equivalent.

Corrosion-Fatigue Strength of J-1h Alloy:

Magnesium sheet alloys, of the type used in this investigation, are known to be susceptible to stress corrosion. For that reason, it is difficult to devise a corrosion-fatigue test that is fundamentally significant, since stresses significant in fatigue would eventually cause failure under static load. Thus, it would be expected that the corrosion-fatigue strength would depend, to a large extent, on the speed of test. The purpose of the test finally adopted was to determine whether the stress-corrosion effect is accelerated by alternating stresses. Since J-1h alloy was more susceptible to stress corrosion than other alloys, it was chosen for investigation. Tests were run at 1500 cycles per minute.

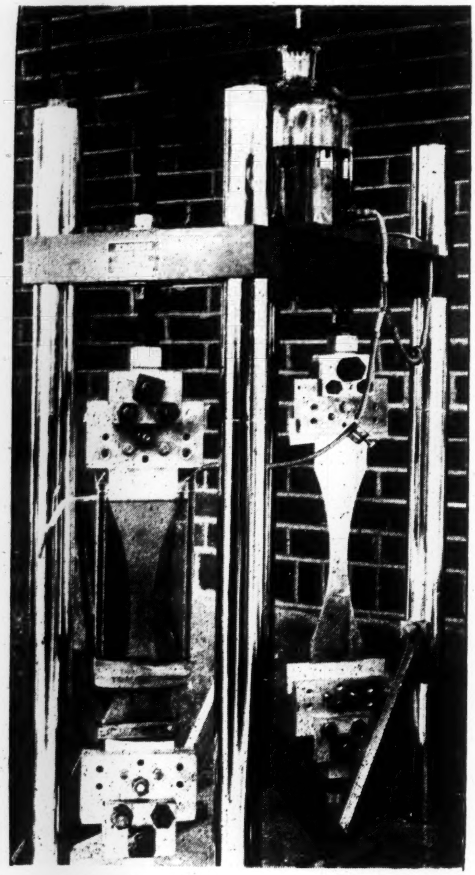

FIG. 16.—Specimen Mounted for Corrosion-Fatigue Test.

Type 5 test specimens were used. These were surrounded with a plastic corrosion cell as shown in Fig. 16. The corroding solution used in the tests is one in common use for studying the susceptibility of magnesium alloys. It consists of 35 g. of NaCl and 20 g. of K_2CrO_4 per liter of distilled water. Enough HCl is added to adjust the pH of the solution to 6.5. When this solution was used in the corrosion cell, it was found that the pH changed slowly because of contact between the solution and the test specimen. Accordingly, the cell was arranged so that solution was removed and replaced by fresh, at a rate which would keep the pH constant.

Figure 17 shows the effect of the solution under static stress and Fig. 18 shows results under repeated stress. Comparison of Figs. 17 and 18 leads to the conclusion that little, if any, acceleration of the stress-corrosion effect is produced by alternating, as compared with static stresses.

CONCLUDING REMARKS

In appraising the evaluation of magnesium sheet alloys given in this report, it should be kept in mind that the reaction of these alloys to repeated stresses is only a part of the larger problem of completely evaluating magnesium alloys as materials of construction. Furthermore, not enough heats of alloys were tested to insure that results are typical.

With these limitations in mind, the following generalizations appear to be warranted:

1. None of the magnesium alloys was

statically as strong as the commonly used aluminum alloys; however, since the magnesium alloys are lighter, it is possible that some designs could provide a comparable strength-weight ratio.

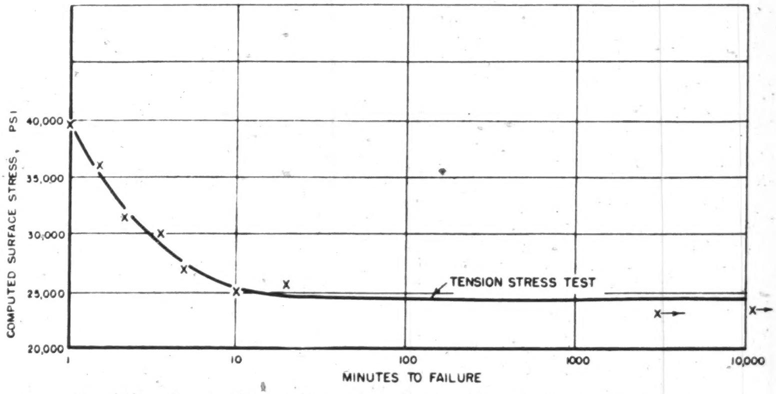

FIG. 17.—Static Stress-Corrosion Curves for 2J-1h As Received.

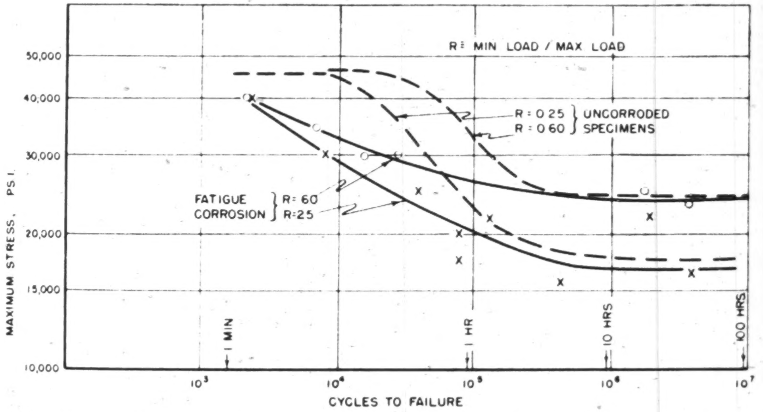

FIG. 18.—Fatigue-Corrosion Curves for 2J-1h As Received. (Type 5 Test Specimens.)
(Specimens 1.000 by 0.064-in. center section, speed 1500 cycles per minute)

2. Relative to static strength, none of the magnesium alloys has as high fatigue strength as unclad 24S-T, for example; however, the fatigue strength compares favorably with the clad 24S-T type alloy.

3. The fatigue strength of magnesium alloys does not appear to be sensitive to orientation with rolling direction.

4. As far as these laboratory fatigue tests show, the notch sensitivity of the various magnesium alloys investigated does not appear to be any greater than that observed in other light metal alloys.

5. While some magnesium sheet alloys are susceptible to stress corrosion, the effect does not appear to be accelerated by repeated stress.

DISCUSSION

MR. G. H. FOUND[1] *(presented in written form.)*—This is a valuable contribution to the field of fatigue testing, particularly because of the diversity of variables studied which may affect the results of axial or direct stress fatigue testing. These data have additional value because they either supplement or substantiate the present available data for direct stressing on magnesium sheet.

We have performed similar direct stress fatigue studies. With two exceptions, our results are in close agreement with the results obtained by these investigators.

These exceptions are the results for FS-1a and 24S-T alloys. In the case of FS-1a, both the static properties in Table III and the fatigue results in Table IV follow in the range for FS-1h. It appears, therefore, that the material referred to as FS-1a in the paper was perhaps mislabeled and is actually FS-1h. Our tests on FS-1a show lower results than for FS-1h. They are, in fact, practically identical with the results for AMC-52S-O, which is the designation of an alloy practically the same as FS-1a which is made by another manufacturer.

Our results for 24S-T of 0.25-in. bare sheet for an R value of 0.25 are 58,000, 40,000, and 27,000 psi. at 10^5, 10^6, and 10^7 respectively instead of 60,000, 52,000, and 51,000 psi. as given in Table IV.

There is an inconsistency between a statement on page 794 under stress corrosion and on page 795 under conclusion 5. The statement on page 794 which says that all of the magnesium sheet alloys which were tested are known to be susceptible to stress corrosion is incorrect. M alloy sheet, which was tested, is known to be not susceptible to stress corrosion.

With regard to conclusion 1, magnesium sheet alloys are being used in service in many designs where a definite static strength to weight advantage is being realized over aluminum. With regard to conclusion 2, a re-examination or redetermination of the fatigue data for 24S-T will modify this statement, especially for the long life range if the data presented in this discussion prove to be correct.

Quantitative or even qualitative use of test-bar fatigue data of the direct stress type alone for predicting serviceability is not advisable, since not only do failures always occur at stress concentrations (of which fabricated sheet structures have many), but fatigue failures often originate where either localized or general bending stresses are superimposed over the axial or direct stresses. Fatigue failures usually originate in such areas as formed regions and regions adjacent to rivet heads where not only do unpredictable stress concentrations exist, but the bending type of loading is also found. The comparison between the fatigue properties of magnesium and aluminum sheet alloys in bending is somewhat different from the comparison for direct-stress fatigue testing. This will also be shown in another paper[2] on fatigue properties of magnesium and aluminum alloys presented at this session.

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[1] Assistant Director, Product Development Div., Magnesium Laboratories, The Dow Chemical Co., Midland, Mich.

[2] See p. 715.

MR. JOHN M. LESSELLS.[3]—Mr. Peterson, who has taught us to consider strength-reduction factors in the fatigue of steel, should be pleased to learn that this same reasoning is now being applied to magnesium alloys.

As regards the path of a fatigue crack in steel, it is generally accepted as being transcrystalline, and only in exceptional cases is the fracture intercrystalline in nature. It would be interesting to hear from Mr. Jackson if this is also true for magnesium alloys, especially for cases of stress concentration.

MR. R. E. PETERSON.[4]—A comparison has been made with theoretical factors, and I should like to remark that I believe such a comparison should be made only if the stress is completely alternating.

If the stress varies sinusoidally between two limits, it can always be broken into an alternating stress component and a steady stress component:

$$S_v = \frac{S_{\max} - S_{\min}}{2}; \qquad S_o = \frac{S_{\max} + S_{\min}}{2}$$

The accompanying Fig. 19 shows the usual way of plotting such stress components.

If we consider the case of zero to maximum, the authors' factor would be AB divided by CE. I should like to suggest that the results be evaluated by use of a factor DF over DE.

MR. R. L. TEMPLIN.[5]—The authors have stated in their paper, (see p. 790), that "This is also true in aluminum alloys where fatigue tests indicate that the statically stronger, but less ductile, 75S-T type alloy has no better fatigue strength than 24S-T." I cannot agree with this statement, because fatigue tests of rolled rod of these two alloys, made using the R. R. Moore type rotating-beam test, give an endurance limit of about 18,000 psi. for 24S-T and an endurance limit of about 22,500 psi. for 75S-T. Fatigue tests of these materials in the form of alclad sheet however give endurance limit values of about 12,500 psi. for both Alclad 24S-T sheet and Alclad 75S-T sheet. These fatigue tests were made using the repeated flexure type of fatigue machine previously described before this Society.[6] In the case of the alclad sheet we think that the fatigue strength of the cladding is a major influence in determining the fatigue strength of the materials.

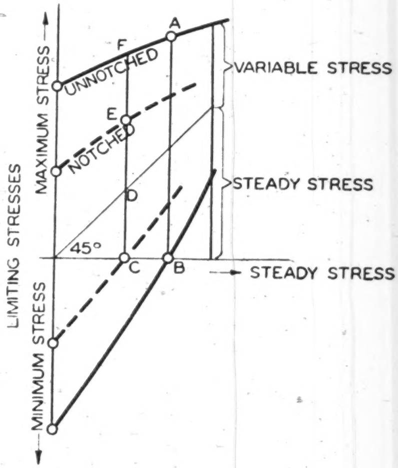

FIG. 19.—Plot of Stress Components.

MR. L. R. JACKSON (*author's closure*).—I appreciate the helpful discussion from the various men who have commented on this work and wish to answer some of the points brought out in their remarks.

Mr. Found's comments can be answered as follows:

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[3] Associate Professor of Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

[4] Manager, Mechanics Div., Westinghouse Research Labs., Westinghouse Electric Corp., East Pittsburgh, Pa.

[5] Assistant Director of Research and Chief Engineer of Tests, Aluminum Research Labs., Aluminum Company of America, New Kensington, Pa.

[6] R. L. Templin, "The Fatigue Properties of Light Metals and Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 367 (1933). See Figs. 2 and 3.

1. His suspicion that the sheet labeled as FS-1a in Tables III and IV should be FS-1h is probably justified. We accepted the manufacturer's designation on this lot of material.

2. As shown in the caption at the head of the section on corrosion fatigue, remarks in that section were intended to apply only to the alloy J1-h so there is no discrepancy.

3. The test results on bare 24S-T sheet, quoted by Mr. Found, are interesting and may indicate a difference between lots of material; however, in connection with other investigations, we have made extensive tests on bare 24S-T sheet and see no reason for adopting the values suggested by Mr. Found in preference to our own; it therefore, does not appear to be necessary to alter conclusion 2.

4. We agree with the idea expressed by Mr. Found that stress concentrations occurring in actual structural parts may alter conclusions based on simple test pieces.

In answer to Mr. Lessells' question as to whether fatigue cracks in magnesium alloys are transcrystalline or intercrystalline, I can say that we examined in detail only those cracks that were obtained under corrosion-fatigue conditions. Under these conditions, we observed both transcrytalline and intercrystalline cracks.

Mr. Peterson's suggestion as to the proper method of comparing stress concentration is exceedingly timely. In this paper we adopted the convention of computing the stress concentration as the ratio between maximum stresses for points on curves taken at the same stress ratio. This method of computation assigns some responsibility to the steady stress in determining the effective stress-concentration factor. Mr. Peterson suggests that the steady stress has little, if any, effect. In the course of extensive investigation, on a wide variety of materials, we have computed stress-concentration factors by both methods, and have been unable to come to any clear-cut decision as to which method is correct or whether either method is correct for all materials. We believe the point is an important one, however, and hope that it can be clarified in the near future, although we are of the opinion that both the convention used by us and the one proposed by Mr. Peterson are oversimplified.

Referring to Mr. Templin's remarks at long lifetimes, it seems to be fairly well established that 75S-T is stronger than 24S-T, but at short lifetimes, which are really the most important, the 24S-T is the stronger of the two. There seems to be quite a fair amount of data in agreement on that.

I wonder whether it is correct to make the comparison at the endurance limit, when a shorter lifetime is really the most important part of the curve.

FATIGUE TESTS OF RAIL STEEL UNDER COMPRESSIVE STRESS*

BY R. S. JENSEN[1] AND H. F. MOORE[1]

SYNOPSIS

Occasional failures of railroad rails occur as fatigue fractures in the fillet joining the head and the web under a range of stress from compression to tension equal to about 20 per cent of the compression. There are also diagonal shearing stresses.

Fatigue tests were made on T-shaped specimens cut from a rail web under cycles of stress varying from compressive stress to tensile stress equal to 20 per cent of the compressive stresses. The testing machine used was a vibratory machine.

The fractures for such specimens started on the compression side of the specimen and the compressive stresses were about 77 per cent higher than the stresses in specimens subjected to cycles of completely reversed bending stress.

Tests of specimens with stamped letters at the critical section on the compression side resulted in reduced stresses at fracture.

Although shot peening sets up longitudinal compressive stress in the unstamped specimens, the fatigue strength of shot-peened specimens was approximately 32 per cent greater than for specimens in which the surface of the compression side was left in the as-rolled condition.

In the joint investigation of the breakage of railroad rails now in progress at the University of Illinois, a type of failure which has become of considerable importance is being studied. Some rails fail by a longitudinal fracture along the fillet joining the head of the rail to the web, as is shown in Fig. 1. These failures occur on the side of the rail in which the major stress in the web is a vertical compressive stress. Based on a large number of field tests of stress pattern in the region where these "web separation" failures occur, it is reported by G. M. Magee,[2] Research Engineer for the American Railway Engineering Assn., that in service a common range of stress in the head-web fillet is from a maximum compressive stress to a tensile stress 20 per cent as great. Together with these tensile and compressive stresses, there are necessarily shearing stresses inclined to the axis of the rail. The amount of fatigue testing which has been done on metals under a range of stress in which compressive stress is greater than the tensile stress is small,[3] and, so far as the authors know, no fatigue tests have been made under the range of stress used in the tests described in this paper.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] Special Research Associate in Engineering Materials and Research Professor of Engineering Materials (Emeritus), respectively, University of Illinois, Urbana, Ill.

[2] *Proceedings*, Am. Railway Engineering Assn., Vol. 46, pp. 660–732 (1945).

[3] H. R. Thomas and J. G. Lowther, "Fatigue Failure Under Repeated Compression," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 421 (1932). Tests mainly on structural steel.

S. W. Lyon, "Mechanical Properties of Rail Steel," *Proceedings*, Am. Railway Engineering Assn., Vo. 42, p. 728 (1941). Fatigue test results of rail steel specimens under cycles of stress ranging from a small tensile stress to a large compressive stress, tests under axial loading (push-pull tests). This report is also printed as Reprint No. 21 by the Engineering Experiment Station, University of Illinois, Urbana, Ill.

TEST SPECIMENS

The test specimens for this study were machined from vertical strips cut from the web of a 112-lb. rail. In order to insure failure starting in a region of high compressive stress, as it has occurred in a rather large number of rails in service, the specimens were designed with a T-shaped minimum cross-section as shown in Fig. 2. From the rectangular strip cut from the rail, part of the metal was milled out on both sides leaving the stem of the T in the middle. These specimens were tested as cantilever beams in partially reversed flexure, with the maximum tensile stress on the top surface of the stem equal to 20 per cent of the maximum compressive stress. The shape and dimensions of the specimen limited the maximum tensile stress on the *bottom* of the specimen to 57 per cent of the maximum compressive stress on the top surface of the specimen; and, in all fatigue failures during the tests on specimens of this shape and size, the fatigue crack started on the top surface of the T, on the "compression side" of the specimen.

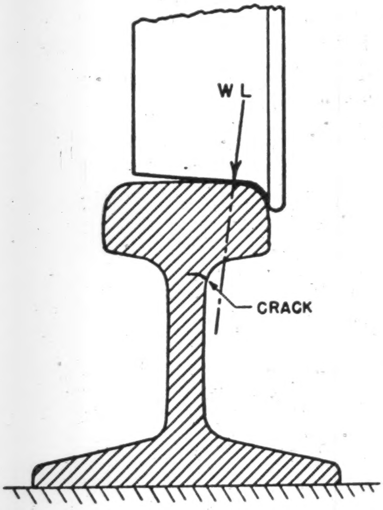

FIG. 1.—Fatigue Crack in the Head-Web Fillet of a Railroad Rail.

WL indicates the wheel load on the rail.
Note that the crack is on the compression side of the web.

In some of the specimens the $\frac{1}{4}$-in. top surface of the stem was left in the as-rolled condition, in some specimens it was left in the as-rolled condition with the imprint of a steel-stamped number at the critical section, and in still other cases the specimen was shot-peened over both top and bottom surfaces. Two sets of specimens were tested with the critical section in the shape of a rectangle (sides of flange of T-section milled off) under cycles of reversed flexural stress. One of these sets had polished surfaces at top and bottom, and the other had one surface left in an as-rolled condition. In all cases the thickness of the specimen was very nearly the full thickness of the web of the rail.

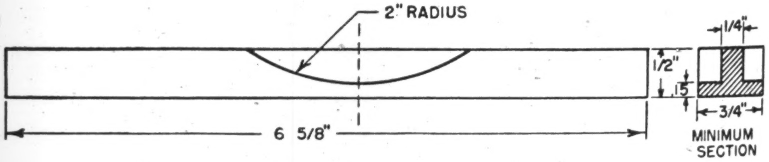

FIG. 2.—T-Section Flexural Fatigue Specimen.

METHOD OF TEST

All specimens were tested in two vibratory type fatigue machines built in the shops of the Talbot Laboratory. Figure 3 shows these machines. Before testing, each specimen was calibrated by dead weights as a dynamometer which indicated bending moment and consequently

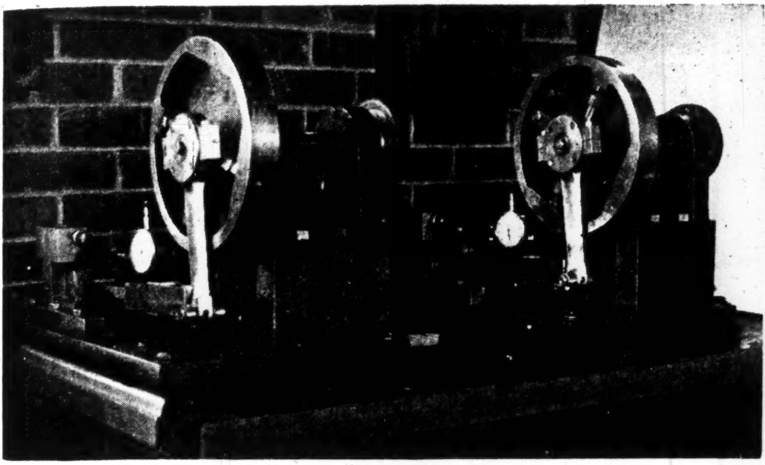

FIG. 3.—Vibratory Flexure Testing Machines.

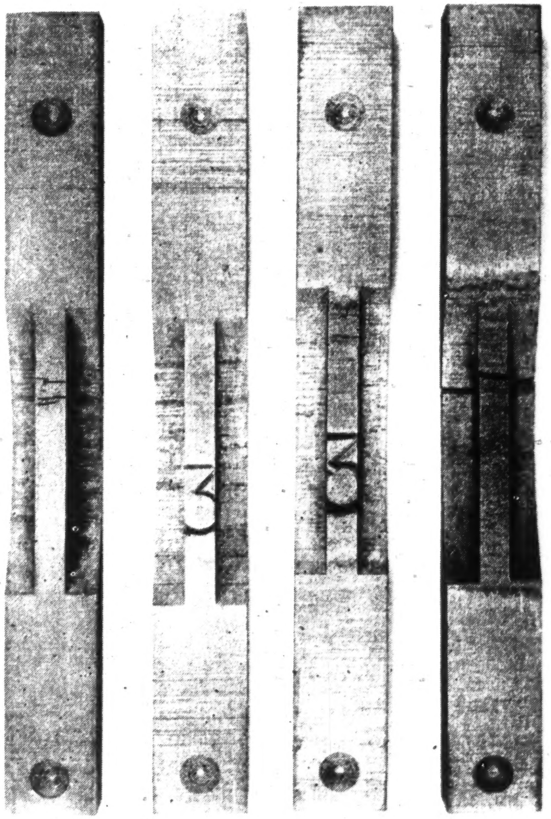

FIG. 4.—Etched T-Section Fatigue Specimens.

The stamped specimens in this figure ran out to 10,000,000 cycles of stress without failure.

nominal stress for any range of amplitude of vibration which may be set up by the variable-throw crank of the testing machine. The machines were run at 800 rpm., and by comparing measurements of the deflection of the specimen when the machine was running and when it was turned over by hand, it was found that the difference in deflection of specimen was very small. During all tests, the constant ratio of maximum tensile stress to maximum compressive stress was maintained at −0.20 by stopping the machine at intervals, taking readings of deflection of specimen, and, if necessary, adjusting the throw of the crank or the tilt of the vise holding the specimen.

Results of Tests

For the tests running to 1,000,000 or more cycles of stress, the stress in the specimen probably was below the yield strength of the steel, the modulus of elasticity of the steel was not appreciably changed, and the deflection of the specimen may be regarded as a reliable measure of the stress. For the tests running under 500,000 cycles of stress, the actual stress is somewhat less than that indicated by the deflection of the specimen.

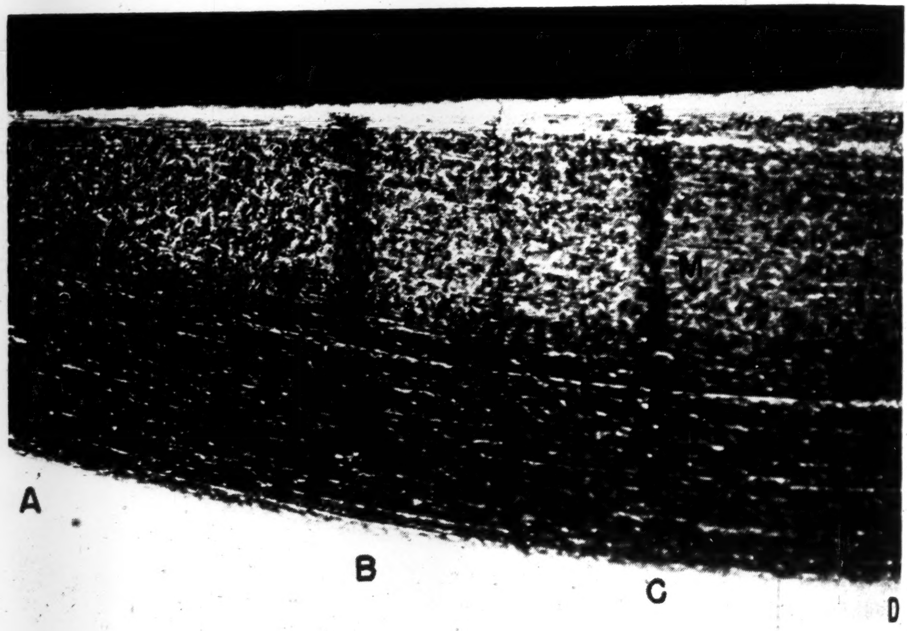

Fig. 5.—Side View of Crack in Shot-Peened T-Section Fatigue Specimen (×8.6).

M and *M* are pencil lines drawn on specimen to aid in locating the crack when viewed through the microscope. *ABCD* locates the upper edge of the (curved) flange of the T-section specimen.

Photograph by C. W. Dollins

The fatigue crack starting on the compression side of the specimen spread much more slowly than a crack starting on the tension side. The following tentative explanation of the progress of a fatigue crack starting in a region of high compression is offered. The crack starts under shearing stress, probably diagonal in direction, but once formed, the crack becomes a very effective stress raiser, and the following tensile stress of 20 per cent of the maximum compressive stress may well be increased to 50 or even to 60 per cent. This is sufficient to cause the crack to spread and to follow at right angles to the direction of the maximum tensile stress, which, incidentally, is in the same direction as that of the maximum compressive stress. However, under this relatively small tensile stress, the rate of spread of the crack would be slow. Figure 4 shows top surface views of fatigue cracks and Fig. 5 shows a side view of a crack.

Not only is the progress of the crack slow in a region of high compression, but, especially in the case of a T-shaped specimen, it may almost, if not entirely, cease to spread when it reaches the upper edge of the flange—if, as was the case for these tests, the testing machine is of the constant-deflection type. As the crack progresses into the specimen, the cross-section below the crack and the moment of inertia are diminished. Hence, for a given deflection of the specimen, the stress at the extreme fibers, which are then at the bottom of the crack and at the bottom of the specimen, is diminished,—so greatly diminished when the crack gets down to the upper edge of the flange of the specimen that the stress may well be lower than the long-time endurance limit of the metal, even allowing for the stress-concentration due to the crack.

As a result of these considerations, the criterion of failure in T-shaped flexure specimens in which the crack starts on the compression surface may well be taken as the first appearance of a crack, and checked by watching to see whether the crack spreads.

Several of the specimens tested developed more than one crack on the top surface. These additional cracks were usually small and often could not be seen until the specimen had been etched with hot hydrochloric acid. Two such etched specimens are shown in Fig. 4, and a magnified side view of one unetched specimen is shown in Fig. 5.

Figure 6 shows the results of the fatigue tests of specimens tested under range of stress from maximum compressive stress to a tensile stress 20 per cent as large. The middle *S-N* diagram shows the results of the tests of specimens with top surface left in the as-rolled condition, and the endurance limit under 10,000,000 cycles of stress is seen to be 59,000 psi.

EFFECT OF STAMPED LETTERS

While stamped letters on rails are located along the middle of the height of the web rather than at the head-web fillet, where the troublesome service failures have occurred, the effect of stamped letters on the fatigue strength of the steel is of interest. A set of T-shaped specimens was prepared with a stamped number at the surface of the specimen at the minimum section. Two such specimens are shown in Fig. 4. Since the stamped letters were irregular in shape, some with a sharp V-notch and some with a rounded notch, and since the depth of stamping varied from 0.014 in. to 0.60 in., the calculated stresses for these specimens were based on the full thickness of the specimen. As might be expected, there was considerable scatter of results for stamped specimens, as is shown in Fig. 6 in which the lower *S-N* diagram is for the stamped specimens. The endurance limit under 10,000,000 cycles of stress was about 51,000 psi. as compared with the endurance limit of 59,000 psi. for the as-rolled specimens without stamped numbers.

EFFECT OF SHOT PEENING

This study of the strength of rail steel furnished an opportunity to try an experiment on the strengthening (or weakening) effect of shot peening on regions

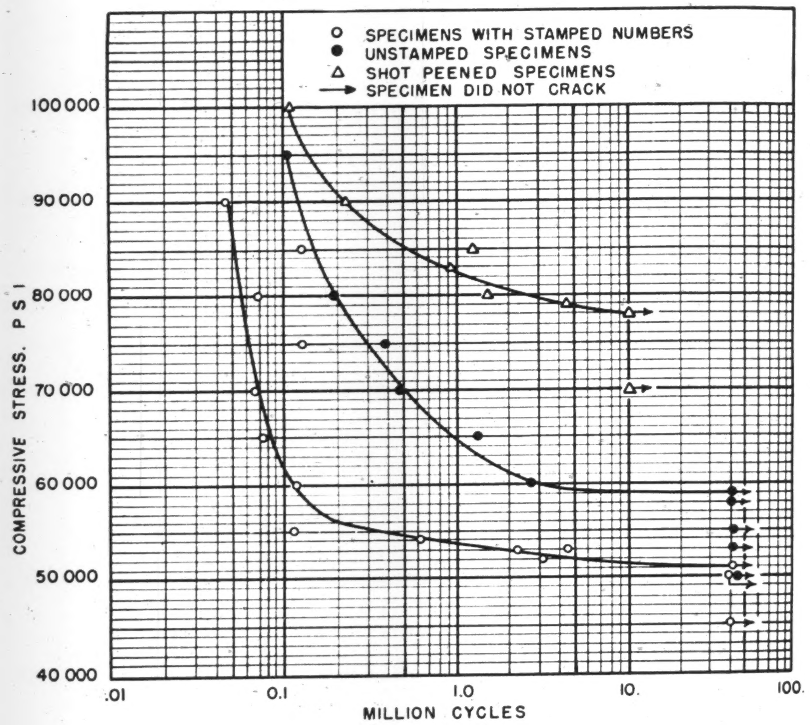

FIG. 6.—*S-N* Diagrams for Fatigue Tests of T-Section Specimens in Flexure.

Maximum tensile stress during a cycle of stress was 20 per cent of the maximum compressive stress.

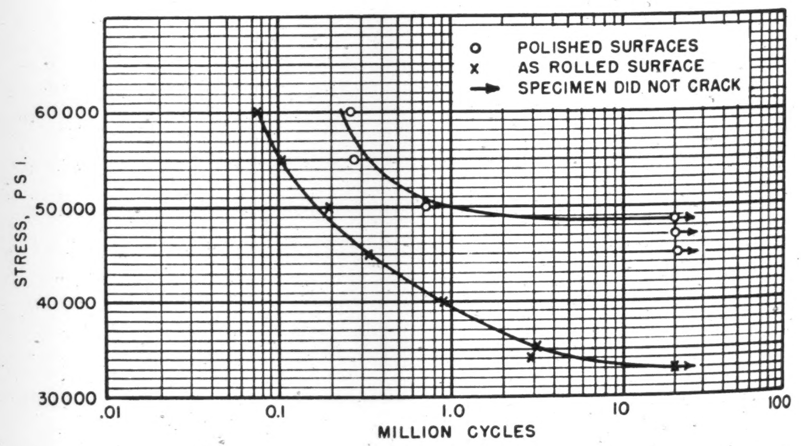

FIG. 7.—*S-N* Diagrams for Fatigue Tests of Rail Steel Specimens Under Completely Reversed Flexural Stress.

in steel parts where the dominant stress was compressive. Shot peening produces its effects by the plastic deformation of metal, and after a part is shot-peened there remains a complex system of residual stresses in it. In the fatigue specimens cut from the web of a rail, shot peening left longitudinal residual compressive stresses in the specimen, and it seemed that the longitudinal stresses due to shot peening added to the compressive stresses caused by the repeated cycles of vibratory loading might act to reduce the fatigue strength of the metal on the compressive side. If the *transverse* residual stresses and the *vertical* residual stresses set up close to the surface were considered as well as the longitudinal residual stresses, this weakening effect might well be considered as somewhat diminished. Moreover, the process of cold working changes the alignment and the shape of the crystalline grains, and, in many cases, increases the resistance of the metal to slip and cracking.

Eight T-shaped specimens from the same rail as the other T-shaped specimens were shot-peened by the American Foundry Equipment Co. of Mishawaka, Ind., and tested under cycles of stress ranging from maximum compressive to 20 per cent as great tensile stress. In Fig. 6 the upper *S-N* diagram shows the results of the tests of these shot-peened specimens. The endurance limit under 10,000,000 cycles of stress is 78,000 psi. for the shot-peened specimens as compared with 59,000 psi. for the as-rolled specimens, a gain of 32 per cent due to shot peening. Whatever the explanation, the shot-peened specimens were distinctly more resistant to fracture than were the corresponding specimens tested with surface as-rolled.

EFFECT OF POLISHING

For a comparison of the fatigue strength in regions of high compressive stress with the fatigue strength in regions of high tensile stress, two groups of specimens were tested under cycles of completely reversed flexural stress. The critical cross-section of these specimens was rectangular rather than T-shaped. For one set of specimens the surface was left as-rolled, and for the other the surfaces were polished. As shown in Fig. 7, the polished specimens developed an endurance limit of 48,600 psi. under 10,000,000 cycles of stress, while the as-rolled specimens developed an endurance limit of 33,400 psi. That is, the polished specimens showed 46 per cent greater fatigue strength under reversed stress than the specimens with as-rolled surface. The specimens with as-rolled surface tested under cycles of flexural stress ranging from maximum compressive stress to tensile stress 20 per cent as great developed an endurance limit under 10,000,000 cycles of stress which was 77 per cent higher than that for the corresponding specimens tested under cycles of completely reversed stress. Unfortunately, time did not permit the extension of the tests to a study of the effect of range of stress during a cycle on as-rolled, polished, and shot-peened specimens.

SUMMARY

1. In this paper are reported the results of fatigue tests of rail steel in which the range of stress varied from maximum compressive stress to a tensile stress 20 per cent as great. In all specimens tested under this range of stress, the fatigue crack started on the compression side of the specimen.

2. The specimens of rail steel tested under this range of stress showed an endurance limit under 10,000,000 cycles of stress, 77 per cent higher than for corresponding specimens tested under cycles of completely reversed stress.

3. Under a stress range from maximum compressive stress to tensile stress 20 per cent as great, a marked reduction of fatigue strength was caused for specimens with stamped numbers at the critical section of the compressive surface of the specimen.

4. Shot peening of specimens raised the fatigue strength 32 per cent above fatigue strength of as-rolled specimens, for this range of partially reversed stress.

DISCUSSION

Mr. J. O. Almen.[1]—The fatigue tests reported by Jensen and Moore are titled "Fatigue Tests of Rail Steel Under Compressive Stress." There is no doubt that portions of the specimens were stressed in compression, but the failures, when failures occurred, were the result of tensile stresses. This could be readily ascertained by measuring the residual stress in one of the specimens after it had been subjected to a large number of stress cycles, but before failure occurred. Such measurements would show that the material in the vicinity of the fractures had developed residual tension because of local yielding of the material under the repeated compressive stress. The specimen would therefore be loaded in tension at the point of fracture whenever the external load was removed. Hence the fracture would actually be caused by repeated tensile stresses.

In conducting these tests, the experimenters attempted to determine the behavior of the material under arbitrary conditions of specimen shape and specimen load. The data obtained will, of course, measure the fatigue strength of the material under the conditions that prevailed during the test, but they will not necessarily reflect the behavior of the same material when formed into another shape of specimen and subjected to a different loading sequence. The reason for this variation in material behavior will be understood when it is appreciated that the causes of fatigue failure are more frequently found in (1) the shape of the specimen or machine element, (2) the methods that were used in fabrication, (3) the load sequence that is applied, and (4) the operating environment. These factors, rather than the material, dominate the behavior of the specimen.

For predicting the behavior of structural parts or machine elements when it is important that we obtain the maximum work from the material used, we must conduct tests on the finished machine element in its actual operating environment. This means that the specimen must be tested as an integral part of an operating machine in normal service.

The second best approach is, of course, to conduct laboratory fatigue tests on the finished machine element under "simulated" service conditions. This can be done with good accuracy if the service history of similar machine elements is known, because we can then more nearly "simulate" service conditions.

There is, of course, some advantage in conducting fatigue tests on arbitrary laboratory specimens when data from the actual machine parts are not obtainable, but it is very dangerous to assume that the data obtained from the specimen tests can be applied in a quantitative sense to the structural parts made from the same material. This is true for all kinds of machine elements whether or not they are prestressed. I have spent about thirty years conducting fatigue tests on laboratory specimens and on machine elements in the laboratory and as integral parts of normally operating machines. I have become quite pessimistic regarding the use that is normally made of laboratory fatigue data that are obtained from arbitrary fatigue specimens, because I rarely find correlation between such data and the data obtained from the machine elements in actual service.

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[1] Head, Mechanical Engineering Dept., Research Labs. Div., General Motors Corp., Detroit, Mich.

It must be appreciated that my approach to the problem of fatigue is somewhat different from that of the authors. My experience has been accumulated in industries in which it is necessary that we obtain the maximum work for every pound of material that is used. We may not overcome the shortcomings of our experimental methods by large factors of safety.

CHAIRMAN R. E. PETERSON.[2]—In addition to making comparisons of maximum stress it is often important to consider range of stress. For the data given, the range of stress values are:

1. As-rolled, compression to 0.2 tension	59 000 × 1.2 = 70 800 psi
2. As-rolled, completely reversed	33 400 × 2 = 66 800 psi.
3. Polished, completely reversed	48 600 × 2 = 97 200 psi.
4. Shot-peened, compression to 0.2 tension	78 000 × 1.2 = 93 600 psi.
5. Stamped numbers, compression to 0.2 tension	51 000 × 1.2 = 61 200 psi.

Note that the range of stress is about the same for the as-rolled specimen series 1 and 2. This is also the case if we compare 3 and 4. The value of the work can be greatly increased by making tests of shot-peened specimens subjected to completely reversed stress and polished specimens subjected to compression to 0.2 tension.

MR. JOHN N. KENYON.[3]—The statement has been made that the fatigue test is not a true service test. Do we have any laboratory technique that actually simulates all service conditions? It seems to me that while the fatigue test does not give all the answers, it represents service conditions more nearly than the other physical tests—such as the tension test—upon which so much reliance is placed.

MR. J. B. KOMMERS[4] *(by letter)*.—This paper is especially interesting because it discusses tests on range of stress in which the maximum stress is compressive. It is the writer's opinion that our knowledge of range of stress is incomplete even for cases in which the maximum stress is tensile.

In 1927, Moore and Kommers[5] suggested a modified Johnson-Goodman formula for range of stress, when the maximum stress is tensile, as follows:

$$S_{\text{max}} = \frac{S_{-1} \times 3}{2 - r}$$

in which

S_{max} = maximum unit stress in the cycle,
S_{-1} = endurance limit for completely reversed stresses, and
r = ratio of minimum to maximum stress in the cycle. (r is negative when the stress is reversed.)

In the present tests, the endurance limit for completely reversed stresses was 33,400 psi. in the as-rolled condition. When the minimum stress was tensile and 20 per cent of the maximum stress, the endurance limit was 59,000 psi.

Using the value for completely reversed stress of 33,400 and the value of r equal to -0.2, the computed value of the maximum unit stress is:

$$S_{\text{max}} = \frac{33{,}400 \times 3}{2 + 0.2} = 46{,}000 \text{ psi.}$$

Because the actual maximum stress for this value of r was found to be 59,000 psi., there is an indication here that a cycle with maximum stress compressive is not as dangerous as one with maximum stress tensile.

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[2] Manager, Mechanics Div., Westinghouse Research Labs., Westinghouse Electric Corp., East Pittsburgh, Pa.

[3] Research Engineer, Columbia University, New York, N. Y.

[4] Professor of Mechanics, University of Wisconsin, Madison, Wis.

[5] H. F. Moore and J. B. Kommers, "The Fatigue of Metals," McGraw-Hill Book Co., Inc., New York, N. Y. (1927). See p. 185.

If further tests should show that such behavior under compressive stress may be expected, to what extent would this invalidate the conclusion of H. J. Gough[6] that it is shear stress that initiates fatigue failure?

It would probably be of interest for the authors to report the tensile properties of the steel used in their investigation.

MR. H. F. MOORE (*author*).—I cannot agree with Mr. Almen's broad statement, "The failures, when failures occurred, were the result of tensile stress. This could be readily ascertained by measuring the residual stress in one of the specimens after it had been subjected to a large number of stress cycles, but before failure occurred. Such measurements would show that the material in the vicinity of the fractures had developed residual tension because of local yielding of the material under repeated compressive stress."

In the case of the long-time tests of the unpeened specimens, the maximum compressive stress was not above the yield strength of unpeened rail steel (about 70,000 psi.). In the peened specimens the compressive repeated stress was above the yield strength of unpeened rail steel, but probably not above that of the peened metal, and it seems very doubtful whether the cycles of repeated compressive stress would overcome the residual compressive stress set up by shot peening. I cannot agree with Mr. Almen's sweeping statement that fatigue failure is always started by a measurable tensile stress.

In my opinion the cause of the starting of a fatigue crack in the compression surface zone of the specimens is far more likely to be due to the high diagonal *shearing* stress, which is about half as great as the compressive stress. Ever since the classic studies of Ewing, Rosenhain, and Humfrey, [7, 8, 9, 10] made at the British National Physical Laboratory about fifty years ago, the theory that fatigue fracture *starts* approximately in the direction of the maximum computed shearing stress has been very generally held by metallurgists and students of stress and strain, and this matter was very fully treated in the 1933 Marburg Lecture presented before this society by H. J. Gough,[6] then of the staff of the British National Physical Laboratory.

Under some critical shearing stress, slip of laminae takes place along certain atomic planes within crystalline grains of the metal, with much disturbance of material along the planes of slip, and the development of cracks if the shearing stress is severe enough and repeated enough times. Quite possibly there are micro tensile stresses in the disturbed region, but these micro stresses are at present uncomputable, and they bear no known quantitative relation to the measurable and computable stresses, either those due to repeated stress or to residual stress.

Many observations, especially of fatigue fractures under cycles of reversed torsion, have shown cracks which started in the approximate direction of the maximum computed shearing stress, and then as they extended into regions of tensile stress, even of light tensile stress, the shear cracks acted as stress raisers, which increased the tensile stress until it became large enough to govern the direction of the further spread of the crack.

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[6] H. J. Gough, "Crystalline Structure in Relation to Failure of Metals—Especially by Fatigue," 1933 Edgar Marburg Lecture, *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 3 (1933).

[7] J. A. Ewing and W. Rosenhain, *Philosophical Transactions*, Royal Soc., Vol. 193, p. 353 (1899); Vol. 195, p. 279 (1900).

[8] J. A. Ewing and J. C. W. Humfrey, *Ibid.*, Vol. 200, p. 241 (1902).

[9] W. Rosenhain, *Journal*, Iron and Steel Inst., Vol. 67, Part I, p. 335 (1904).

[10] A concise summary of their work is given in J. W. Mellor, "Crystallization of Iron and Steel" Longmans, Green & Co., London and New York (1905).

In Fig. 5 of our paper, the fatigue crack evidently started in the junction region between peened and unpeened metal, and a very short diagonal crack in the approximate direction of the maximum shearing stress is seen. The direct stress along the upper edge of the specimen varied from compression to 20 per cent as much tension at first, and it is doubtful whether the computed tensile stress would have reached much higher values under repeated stress were it not for the stress raising effect of the diagonal shear crack. This stress raising effect might easily increase the tensile stress from 20 per cent of the compressive stress to 60 or even 80 per cent. This intensified tensile stress might then be thought of as the cause of the *spread* of the crack. However, I still adhere to the commonly accepted view that fatigue cracks *start* in a region of computed shearing stress approximating the maximum computed shearing stress at any location. This discussion also answers a question raised by Mr. Kommers in his discussion.

Mr. Almen's picture of the difficulty of using results of conventional fatigue tests as a basis for design of machine parts is admitted to be true in many respects, but I do not believe the picture is so black as he has painted it. We have today considerable reliable information about effect of range of stress, of size effect of specimen, of surface condition of specimen, and of the theoretical and the actual damaging effect of the common stress raisers, such as grooves, notches, holes, fillets, and screw threads, and it is my belief that by considering these, the results of fatigue tests of small specimens may be used as a basis for design. I wish that Mr. Almen would give a somewhat more constructive picture of how he takes account of service conditions which cannot be duplicated in laboratory tests.

MR. ALMEN.—That is a fair challenge. In our discussions, we should, of course, try to be constructive.

It is true that in laboratory testing of machine elements we must try to simulate service conditions. This, however, is not as easily accomplished as many investigators seem to believe, because we rarely know the manner or magnitude of service loading. Many instances can be cited in which wrong answers have been obtained from "simulated" service testing. The difficulty is not confined to fatigue testing but applies to tests of oils, fuels, tires, and paints.

During the war, it became necessary to conduct fatigue tests on an aircraft engine connecting rod that occasionally failed in service. In this case, we were fortunate in having two of the failed specimens from which we could judge the degree of conformity of our laboratory test with service conditions.

It is not difficult to imagine the manner of service loading of a connecting rod in an aircraft engine. This machine element is subjected to push-and-pull loads, upon which are superimposed relatively small bending stresses. Therefore, to simulate service conditions, we would naturally apply the same kind of loads that occur in service. Such tests were applied to approximately thirty connecting rods, and in no instance did the specimen fracture in the region in which fracture occurred in service, even though varying magnitudes of push-and-pull loads were applied. We eventually succeeded in obtaining fractures identical with the fractures that occurred in service by applying small torsional loads without push, pull, or bending.

It is apparent, then, that even though the tests are conducted on the finished machine elements, we are not assured of correct answers by "simulating" service conditions. Our chances for error are, however, greatly reduced as compared to data that are obtained from arbitrarily shaped specimens tested under arbitrary conditions of load. The load sequence that is applied in service varies in an unpredictable manner, and the laboratory operating environment is usually, of course, very different from that which occurs in service.

The principal reason for misunderstandings among investigators in the service strength of machine parts is to be found in the importance that is attached to the weight and cost of the finished machines. We cannot use the same criteria for evaluating materials and designs that are used in heavy machine equipment for extremely light, high-speed machines such as automobiles and aircraft engines. In the former case, it is usually economical to design to low stresses and to use relatively low quality material, whereas in the latter case, we do not have this freedom.

For machine parts in which it is important to design to the absolute minimum of weight, we must not only use clean metals—which, incidentally, is one of the things that can be determined by the usual laboratory fatigue test—but we must exercise rigid control over the fabrication processes, which often are far more important than the quality of the material that is used.

Many of the processes that we are now employing to improve the fatigue strength of machine elements were not readily accepted by our engineers and metallurgists because they came to us from the wrong side of the track. Such processes as pressure rolling of shafts and peening of springs and other machine elements have been known and have been practiced for a great many years, but our designers and metallurgists have given them scant attention because our stress theories are based upon test data obtained from static tests, under which conditions these cold working processes are usually harmful.

I think that we do not pay enough attention to the performance of machine elements in actual service. If we had the means to accumulate data on the fatigue failures that occur in actual service, we would make more rapid progress than can be accomplished by our present procedures.

In spite of the vast amount of laboratory data that has been recorded over the years, we actually have little information that is useful to the designer as indicating the actual operating stresses and the best proportions for machine designs. It is still true that an experienced designer can make a better guess at the required proportions of many machine elements than can be obtained by the most elaborate calculations.

All of this does not justify the blanket condemnation of laboratory tests conducted on arbitrary laboratory specimens under arbitrary conditions of load. Such tests can be useful as indicating qualitative effects, but the data cannot be used quantitatively in machine design. The effect of shape, fabrication processes, and operating environment are so great as usually to dominate the behavior of the machine parts, regardless of material quality. We must continue running tests, but we must learn how to evaluate these tests in terms of the finished machine part. When we follow these procedures, we will find that laboratory specimens very often play a small part in determining the adequacy of our designs.

The situation in regard to the utilization of material is well illustrated by the experiences of the several aircraft engine manufacturers who built engines for military use. When the war began, four of our large manufacturers were prepared to deliver engines that had been developed over a term of years. These engines presumably represented the best possible practice in material and processing. The fatigue capacity of the material had been determined by numerous fatigue tests, and the details of design had been carefully studied for points of stress concentration.

These engines were delivering about 1000 hp. each, which represented their maximum capacity. The development on these engines continued until, at the close of the war, they could, if crowded, deliver almost 2000 hp. The modifications that were made were so small that they would not be discernible upon casual inspection. The changes did not add materially to the weight of the engine; nevertheless, the capacity had been tremendously increased.

We might assume that some errors had been committed in the original design, else it would not have been possible to increase the operating stress, as indicated by the increase in horsepower. However, no one can be charged with errors in this instance. It merely represents the progress that is possible when fatigue data are obtained on the various machine parts in their normal operating environment, subjected to normal operating stress in normal load sequence. When any part failed in service, a correction in design or processing was made for that particular part at the point of fracture. When this weeding-out process is continued for a long enough time, many of the weaker sections are strengthened without adding weight to the sections that were originally sufficiently strong.

This method cannot, of course, be applied to heavy machines because it is cheaper to use more material than is necessary in order that service failures may be avoided. The designer of heavy equipment is just as correct when he designs to low operating stresses as is the engineer who designs aircraft parts to high operating stresses.

Improvement in design and processing of airplane engine parts is not done blindly. When service failures occur, the local stresses at the points of origin of the fractures are obviously too great. A laboratory fatigue test can be devised by which a machine part can be caused to fracture in the same location and with the same character of fracture as occurred in service. This may require a loading technique differing from that originally assumed by the designer, but if the test seeks out the correct point of weakness, it will obviously be representative of the service loading. The stress may now be measured by any of the several convenient strain measuring devices, and corrections in design or processing can then sometimes be evaluated by strain measurements. In all cases, however, it is necessary to conduct fatigue tests on the revised design, either by actual service use or by full-scale laboratory tests as have been described.

MR. C. A. ADAMS.[11]—How did you obtain the 2000-hp. from the airplane engine? You cannot get 2000 hp. from an engine designed for 1000 hp. without doing one of two things—increasing the speed or increasing the compression. Which method was adopted in this case?

MR. ALMEN.—The increased horsepower was obtained principally by increasing the mean effective pressure, which means increased loads as well as increased thermal stresses. There was also some increase in speed, but this increased the inertia forces and hence increased loads.

Another illustration may be helpful to show the gains that can be made from testing full-scale machine elements in actual service and in the laboratory. Some years ago, a well-known automobile required a 14-in. bevel ring gear to drive the car. Today, this car is driven by a ring gear measuring only $8\frac{1}{4}$ in. in diameter, notwithstanding the fact that it must carry higher torques and operate at higher speeds. The 14-in. ring gear was subject to failure in service, whereas service failures today are virtually unknown. This increased load capacity of gears is due to changes in design and in fabrication practice which were indicated by studies of service failures. Laboratory fatigue tests on full-scale parts were devised by which service failures were reproduced in type and in location, enabling the effect of changes in design, processing, and material to be studied. The materials and heat treatments used today are substantially the same as were used before the above improvements were made, because the causes of failure were found in design and processing.

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[11] Consulting Engineer, The Budd Co., Philadelphia, Pa.

The data obtained from such tests as have been described can be applied to similar machine elements used in the heavy industries field far more advantageously than can data that are obtained from arbitrary laboratory specimens.

MESSRS. R. S. JENSEN AND H. F. MOORE (*authors' closure*).—In the tests described in this paper specimens were cut from the web of a rail, and the thickness of the specimen was only a few hundredths of an inch less than that of the web of the rail. The stress pattern shown by tests on rails in actual service, using electric resistance strain gages, showed that under the passage of wheel loads there developed a range of stress in the fillet joining head and web of rail varying from a maximum vertical compression to a vertical tension about 20 per cent as great, with very small longitudinal stresses. In the laboratory test specimens, the compression face of the T-shaped specimen was left as rolled in the unpeened specimens. The compression side of the specimen was, of course, peened in the peened specimens. The fracture developed in the laboratory tests was of the same general type as that developed in service fractures, a crack on the compression side, spreading inward from the surface zone of the rail. These tests seem to the authors to be a pretty fair example of making and testing specimens so as to reproduce the stress conditions found in service.

As for Mr. Almen's general opinion of conventional fatigue tests, the writers agree with him that the results of such tests should not be carelessly used as a basis for design. We differ with him quantitatively in that we believe that by considering stress concentrations, surface conditions, size effects, range of stress and the production in the laboratory of the same general kind of failure as that found in service, such conventional fatigue test results *may* be used as a basis for design with a good degree of confidence.

The authors note Mr. Peterson's suggestion that further fatigue test results would be valuable, they agree with him, and regret that there has not been opportunity to make the tests he suggests.

In response to Mr. Kommers' suggestion, the following range of tensile properties for rail steel is reported from the data secured by the rails investigation at the University of Illinois:

Yield Strength (0.2 per cent offset)	63 000 to 83000 psi.
Tensile Strength	**129 000 to 146 000 psi.**
Elongation in 2 in.	**7 to 10 per cent**
Reduction of Area	**13 to 18 per cent**

FATIGUE OF WOOD AND GLUED-WOOD CONSTRUCTIONS*

By W. C. Lewis[1]

Synopsis

This paper summarizes the experience in the fatigue testing of wood and glued-wood constructions at the Forest Products Laboratory. Test methods are presented which have been developed for making tests of wood and plywood in cantilever bending, in tension parallel or perpendicular to the grain for solid or glued specimens, and on glued laminated specimens in which the glue joints are stressed in shear.

The endurance limit for wood and glued-wood constructions is not indicated by tests that have continued to 30,000,000 cycles in direct-stress tests and to 50,000,000 cycles in cantilever-bending tests; thus, the results of fatigue tests of wood are reviewed in terms of endurance load for specified numbers of cycles rather than for any estimated endurance limits. Unlike the failures of some other materials, failures resulting from fatigue of wood are of the same character and appearance as those developed in static tests.

The development of new glues, new manufacturing techniques, and new products has greatly accelerated the use of wood and wood-base constructions in structural or semistructural parts. Refinements in design methods have resulted in much more closely designed structures and products. These refinements have made necessary the investigation of the fatigue characteristics of wood and glued-wood constructions.

T. M. Barlow in a paper (1)[2] published in 1929 stated that "Fokker's reasons for retaining wood wing construction for monoplanes are summed up in the word 'fatigue.' Claims of new materials to lightness must be reviewed in association with their fatigue limits. Fatigue in properly seasoned wood is unknown." The probable reason fatigue failures were unknown in the wood in the aircraft of that day was that, with the approximate methods of design used, actual stresses were usually lower than those that would cause failure in fatigue during the normal life of an airplane.

Tests in fatigue of wood and glued-wood constructions have shown that they are subject to failures in fatigue the same as any other material. However, the amount of testing has been limited for the following reasons:

1. The lower moduli of wood, as compared to metals, require much greater deformations for a given stress than are possible in most machines designed for metals; so special machines have had to be designed for testing wood.

2. The variability of wood is such that a large number of tests is required for each variable in a fatigue study.

3. The shearing and bearing strengths of wood are low as compared to the tensile strength; so it has been necessary to develop special means of attaching

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] Engineer, Forest Products Laboratory, Madison, Wis.

[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 832.

specimens to the loading members of fatigue-testing machines.

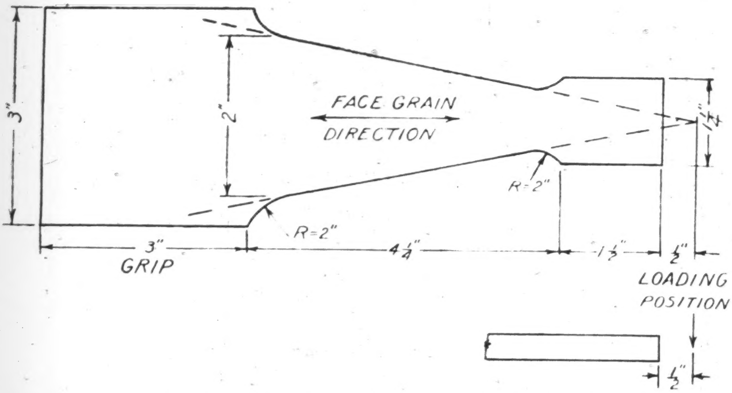
4. The many different commercial species of wood and plywood have required large testing programs to evaluate the basic strength properties; so considerable emphasis has been placed on these studies.

A fatigue-testing program has been in progress in recent years at the Forest Products Laboratory. Some results have been published and more will follow as testing programs are completed. Eleven flat-plate, cantilever-type bending machines were acquired in 1942, and two direct-stress machines (each capable of testing two specimens simultaneously) in 1945. Suitable test specimens and test methods have been developed at the Laboratory for testing wood or plywood cantilever-bending specimens in the flat-plate machines and for making tension-parallel-to-the-grain, tension-perpendicular-to-the-grain, or shear-of-glue-line tests in the direct-stress machines. These specimens and test methods are described in this paper, and results of the fatigue tests of wood and glued-wood constructions performed at the Forest Products Laboratory are presented.

GENERAL CONCEPTS OF FATIGUE AS APPLIED TO WOOD AND WOOD-BASE MATERIALS

Fatigue tests are usually made in one of three types of machines, (*1*) rotating beam, (*2*) cantilever bending, or (*3*) direct stress, where the principal stresses are bending, compression, tension, or shear, either repeated or reversed. Many tests of metals and similar materials have been made on the rotating-beam machines because this type of loading simulates the loading conditions of the material in the finished product. Rotating-beam tests generally are not considered applicable for tests of wood and wood-base materials because only rarely is wood used in rotating members or where the stresses are varied as in a rotating beam specimen. Further, because wood may be considered orthotropic (that is, having three mutually perpendicular planes of elastic symmetry) and because of the annual growth rings, it is usually desirable to have the cross-section of the specimens rectangular.

Flat-plate cantilever-bending fatigue tests are generally considered more applicable for wood and wood-base material than are the rotating-beam tests. Specimens are rectangular in cross-section, usually wider than high, and are loaded at one end and clamped at the other. Care is taken to have a large enough span-depth ratio so that shear stresses are not critical. The span-depth ratio, however, should be kept as low as possible to prevent the natural frequencies of the specimen from approaching the speed of the machine; otherwise, resonant conditions may possibly produce overloads or interfere with the proper operation of the micro-switches. The flat-plate fatigue machines have the disadvantage of being constant-deflection-type machines; so that if the specimens under test yield, the modulus of elasticity changes, or the specimens partially fail, the amplitudes of the stress cycles change. The deformation cycle required to produce the desired variation in stress is set at the beginning of the test, but there is no assurance that this stress cycle remains constant during the entire period of test. If the material under test is not brittle, the test may continue indefinitely without visible failure because of the reduction in the stress cycle. It becomes necessary then arbitrarily to select an end point. Different investigators may, for valid reasons, select different end points, and because of this it is sometimes difficult to compare results.


3"
2"
FACE GRAIN
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R=2"
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GRIP
4 1/4"
1 1/2"
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LOADING
POSITION
1/2"

Fig. 1.—Constant-Stress Type of Specimen (Between Fillets) Investigated for Reversed and Repeated Stress Cantilever Bending Tests.

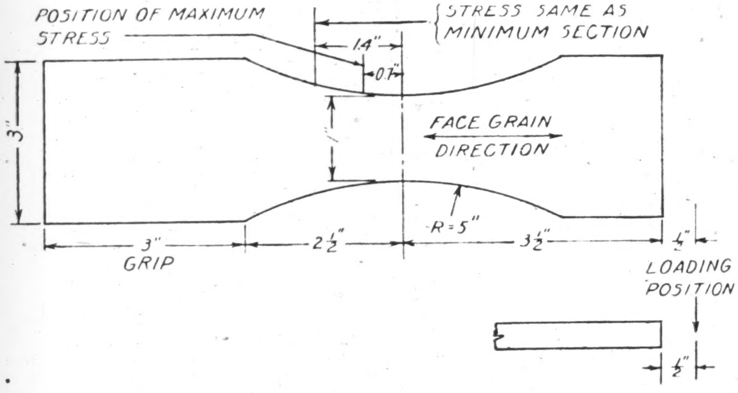
POSITION OF MAXIMUM
STRESS
STRESS SAME AS
MINIMUM SECTION
1.4"
0.7"
3"
1"
FACE GRAIN
DIRECTION
R=5"
3"
GRIP
2 1/2"
3 1/2"
1/2"
LOADING
POSITION
1/2"

Fig. 2.—Reduced-Section Type of Specimen Investigated for Reversed and Repeated Stress Cantilever Bending Tests.

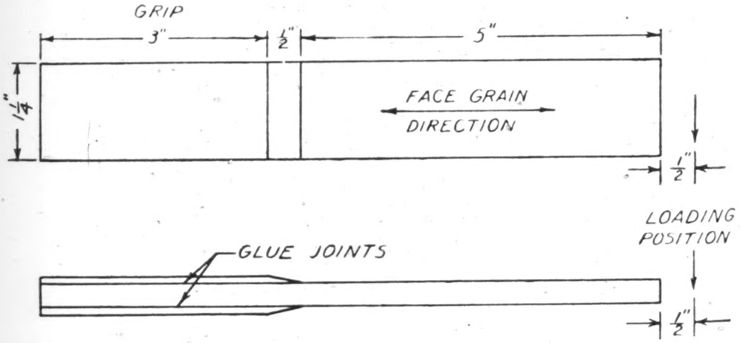
GRIP
3"
1/2"
5"
1 1/4"
FACE GRAIN
DIRECTION
1/2"
LOADING
POSITION
GLUE JOINTS
1/2"

Fig. 3.—Constant-Width Type of Specimen Investigated for Reversed and Repeated Stress Cantilever Bending Tests. Beveled wood strips glued to specimen over gripped portion.

Wood is generally weaker in compression than in tension. This makes it impossible, when conducting fatigue tests in bending with a simple specimen, to obtain any results where the primary failure is in tension rather than in compression, since, in a bending test, the first failure is almost invariably in compression. If the loading cycle is a reversal of stress, the specimen may finally fail in tension because the fibers are first damaged in compression. In a repeated loading cycle the failure consists of a compression wrinkle.

Direct-stress fatigue tests generally are considered to be most applicable for wood and wood-base materials, because the uniformity of stress produced across the section of the specimen allows the data obtained to be used to better advantage in full-sized structures. Any desired stress or variation of stress can be produced by using a properly designed specimen. The only limitation imposed upon a direct-stress fatigue test is governed by the size and capacity of the testing machine. It is possible to repeat loadings in tension or compression or to have either partially or completely reversed tension and compression. By designing the specimen so that tension or compression loadings will produce shear stresses, it is possible to test materials in fatigue in either repeated, partially reversed, or fully reversed shear. If constant force testing machines are used or proper monitoring and shut-off mechanisms are installed on direct-stress testing machines, it is easily possible to test specimens to destruction. Such a procedure has appeared desirable for testing wood and glued-wood constructions.

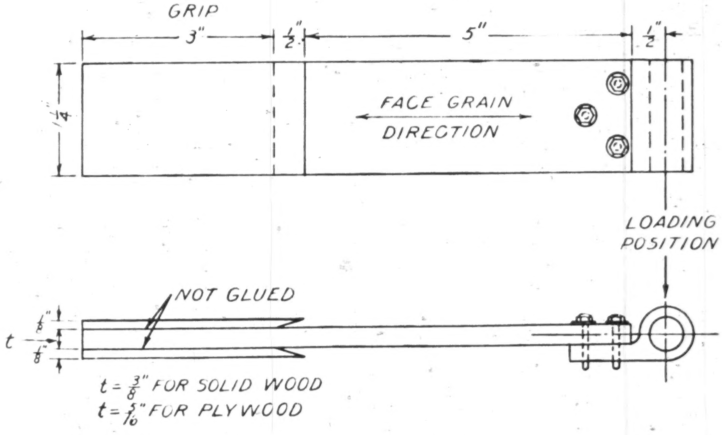

FIG. 4.—Constant-Width Type of Specimen as Used in All Tests Reported. Beveled wood strips not glued to specimen.

METHODS OF TEST DEVELOPED

The design of any specimen for a fatigue test of wood is complicated by the material itself. Radii of curvature in transition sections or fillets must be kept large, otherwise premature failures in shear are likely to result because of concentrations of stress. When the principal stresses are tension or compression parallel to the grain of the wood and there is a stress raiser, such as a hole or a notch present, premature failure in shear is likely to develop at the point of concentration. This was illustrated in a series of static tests of box beams (2) which had holes in the tension flanges. Shear failures started at the edges of the holes when the calculated stress in the tension flanges was approximately 5600 psi., which was approximately 37 per cent of the ultimate strength of the flange. (C. B.

Smith has discussed such stress concentrations in wood mathematically in a report issued by the Forest Products Laboratory (3).) The importance of keeping stress raisers to a minimum for ordinary fatigue work is shown by this example.

Flat-Plate Fatigue Tests:

Development of Specimen.—Several types of fatigue specimens were tried for the flat-plate cantilever-bending test, as reported by W. J. Kommers (4) and shown in Figs. 1 to 4. The constant-stress (between fillets) type of specimen shown in Fig. 1 and the reduced-section type of specimen shown in Fig. 2 were tried because they were similar in shape to specimens used in testing certain plastics. Fillets of different radii were employed at the ends of the uniform strength part of the specimen shown in Fig. 1. It was found that shear cracks formed at the edges of the specimen at the beginning of the fillet nearest the gripped end of the specimen. These failures started at either the top or bottom surface of the specimen and progressed toward the gripped end. In plywood specimens, the shear failures penetrated to the first cross-ply. Similar failures occurred in the specimen shown in Fig. 2. For this reason, the types of specimens shown in Figs. 1 and 2 are not recommended.

The specimen shown in Fig. 3 has a uniform rectangular cross-section with beveled hardwood (maple) plates glued to the gripped end of the specimen. These plates were glued to the specimen to prevent high localized stresses and crushing where the steel grips were applied. Gluing of the beveled pieces to the specimen proper proved impractical because the glue joints failed during the test. The specimen shown in Fig. 4 was finally adopted as the most practical. The beveled plates were not glued to the specimen, but were merely inserted in the grips with the bevel away from the specimen. This specimen was $1\frac{1}{4}$ in. wide, $8\frac{1}{2}$ in. long, and had a uniform thickness. The thickness for plywood was the thickness of the material and for the solid wood it was $\frac{3}{8}$ in.

Test Procedure.—In the flat-plate type of fatigue machine, the specimen itself acts as the dynamometer, for the intensity of the stress is a direct function of the deflection of the specimen. The testing procedure consists of a series of machine adjustments to produce deflections which will give the desired stresses in the specimen. Figure 5 shows a flat-plate fatigue machine with the specimen in place ready for test. This machine has a capacity of 150 lb. and the eccentric is adjustable to provide any desired throw up to 2 in. The electric motor operates at 1790 rpm. The motion of the eccentric E is transferred to the specimen by the connecting rod. The specimen is gripped in the machine at point B and loaded at C, with the distance from the end of the grips (the point of maximum stress) to the center of the pin C, 6 in. To dampen vibrations that might be transmitted to the floor, the machine is bolted to a wood baseboard, which in turn is bolted to a block of concrete. The block of concrete rests on automobile valve springs, which are in compression. The micro-switches F are wired in series with the motor to shut off the machine at the end of the test. A cycle counter is provided on the back of the motor.

When completely reversed stresses are desired, the position of the specimen is adjusted by the nut G until the position of the loaded end of the specimen coincides with the pin C when the cam is in the neutral position. The specimen is loaded at point C with weights sufficient to produce the desired stress in the specimen, the deflection of the specimen noted by means of the dial A, and the weights removed. Next, the connecting rod *D* and specimen are engaged by the pin *C*, and the eccentric *E* is adjusted until the deflection at the extreme position of the cam produces the same reading as when the specimen was loaded with the weights. The dial is removed; the positions of the micro-switches *F* are set with "feeler" gages to a predetermined clearance; the reading of the cycle counter is noted, and the test started.

For partially reversed or repeated stresses, the procedure is similar. To determine the initial position of the specimen with the eccentric in the neutral position, the specimen is loaded to correspond to the desired mean stress, after which the specimen is loaded to the desired maximum stress by additional

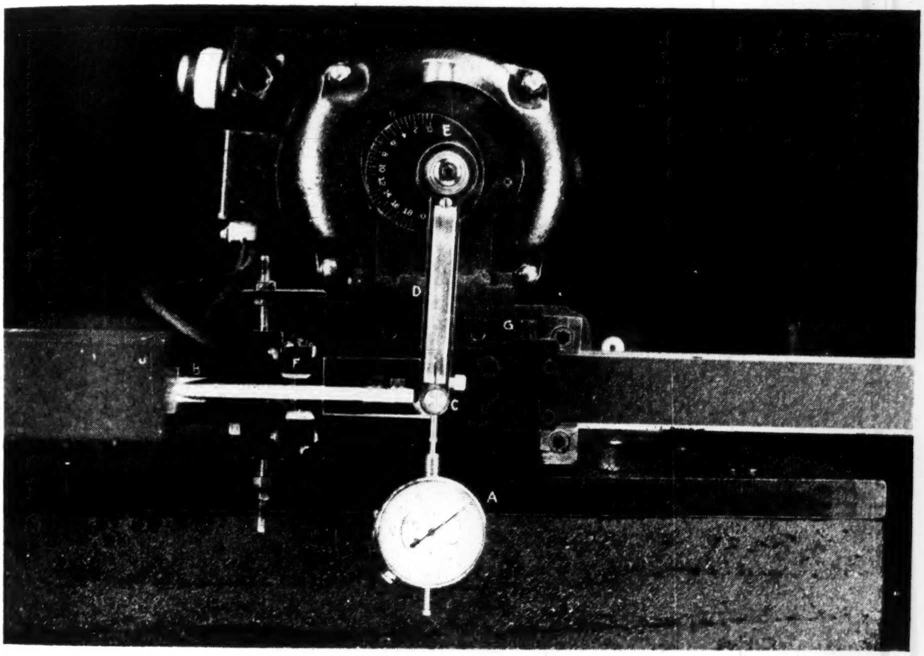

FIG. 5.—Flat-Plate Fatigue Machine Used to Apply the Repeated and Reversed Stresses.

A—Dial to establish deflection to be reached in each cycle
B—Grips
C—Connecting-rod pin
D—Connecting rod
E—Adjustable eccentric to provide desired throw
F—Micro-switches
G—Nut to adjust neutral or mean stress position of specimen.

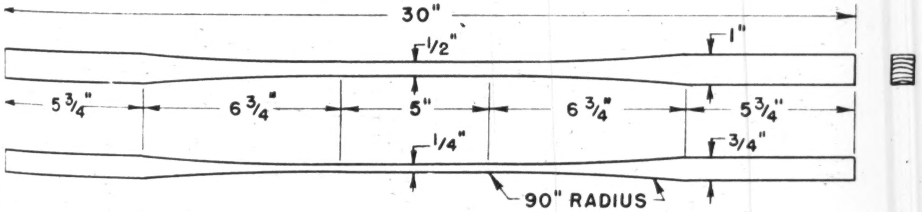

FIG. 6.—Tension-Parallel-to-the-Grain Specimen.

weights and the eccentric adjusted to give a maximum throw equal to that deflection.

One of the necessary mechanisms for any fatigue testing machine is a device that will stop the machine when fracture of the specimen occurs. Several types of shut-off switches that have been used successfully in testing plastics or metals are actuated by the free swing of the connecting rod when the specimen breaks into two pieces. Such a switch is not satisfactory for tests of wood or wood-base materials where the specimen does not completely fracture. It was, therefore, necessary to devise a different type of shut-off that would stop the machine when the outside fibers of either the top or bottom of the specimen failed. Microswitches placed on each side of the specimen at midspan as shown in Fig. 5 were adjusted so that at each extreme of deflection the actuating element cleared the specimen by a small amount. Exploratory tests to determine the appropriate gap or clearance between microswitch and specimen were made with a completely reversed stress cycle. From the results of these tests, it was decided that a change of 0.003 in. in the deflection at midspan would be considered to constitute failure and in subsequent tests the microswitches were set so that this change would shut off the machine. Smaller gaps proved too sensitive to outside disturbances, and larger gaps resulted in failures considerably further progressed than initial failure.

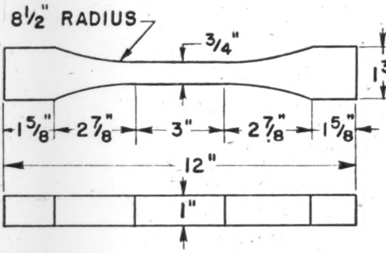

FIG. 7.—Tension-Perpendicular-to-the-Grain Specimen.

Direct-Stress Fatigue Tests:

Development of Specimens.—Specimens and methods of tests have been developed for making direct-stress fatigue tests in tension parallel to the grain, in tension perpendicular to the grain, and on a typical glue joint with the glue line stressed in shear. Drawings of these specimens are presented in Figs. 6 to 8. The tension-parallel-to-the-grain specimen in Fig. 6 is 30 in. long and has a minimum cross-sectional area of $\frac{1}{8}$ sq. in. The minimum section is 5 in. long because in a current series of tests it was desired to include specimens with a glued scarf joint having a 1:8 slope in addition to other specimens. Such a scarf joint, when made across the $\frac{1}{2}$-in. dimension, requires a minimum length of 4 in. The tension-perpendicular-to-grain specimen shown in Fig. 7 has a reduced section in one direction only; the thickness is uniform. The glue-shear specimen[3] shown in Fig. 8 was designed to obtain information on the fatigue behavior of a glued joint when stressed in shear.

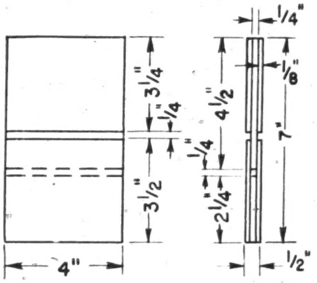

FIG. 8.—Laminated Glue Shear Specimen.

A difficulty in making direct-stress fatigue tests of wood is the proper gripping of the specimens. Wedge grips as used in many static tests are obviously not applicable; and, if bolted connections are used, the bolt holes tend to enlarge and the wood to split as the test progresses. This difficulty was resolved satisfactorily by gluing steel plates to the ends of the specimens. Thin sheets of yellow birch or walnut veneers with the grain parallel to the direction of applied load are glued to each face of the plates using one of the better commercial metal-to-wood adhesives in a hot press. The plates with the veneer attached are then glued to the specimens, using a cold-setting resorcinol resin glue. This method offers several advantages over gluing the plates directly to the specimens. After completion of one test, the plates are removed, turned over, and glued to another specimen. This enables the plates to be used twice for each gluing in a hot press. Because the specimens are not subjected to high temperatures in setting the glue with consequent drying, less conditioning is required to bring them to a proper moisture content for test. The plates are reusable so the cost is not excessive.

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[3] This specimen is similar to a specimen developed by D. G. Radway and N. V. Poletika of Curtiss-Wright Corp. and discussed in their Report No. WE-11-M-10, June 7, 1944.

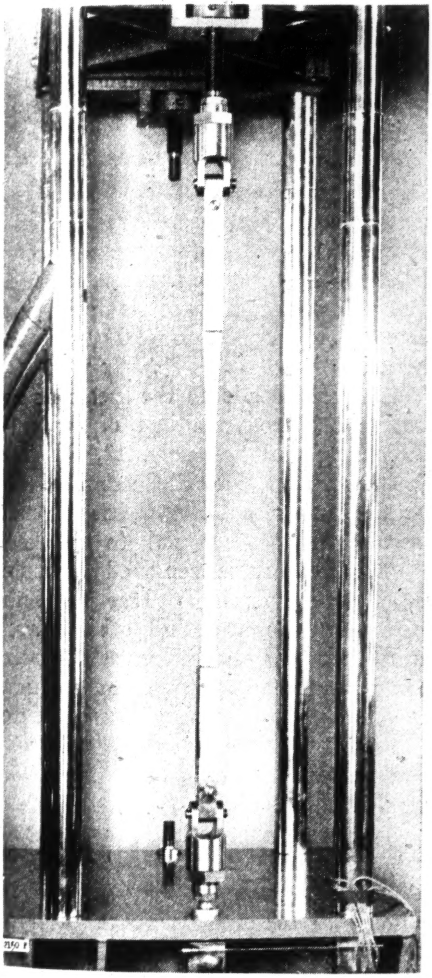

FIG. 9.—Tension-Parallel-to-the-Grain Specimen in 4000-lb. Direct-Stress Fatigue Machine, Showing Self-Aligning, Pin-Connected Fittings and Steel Plates Which Transmit the Load from the Loading Screws to the Specimen.

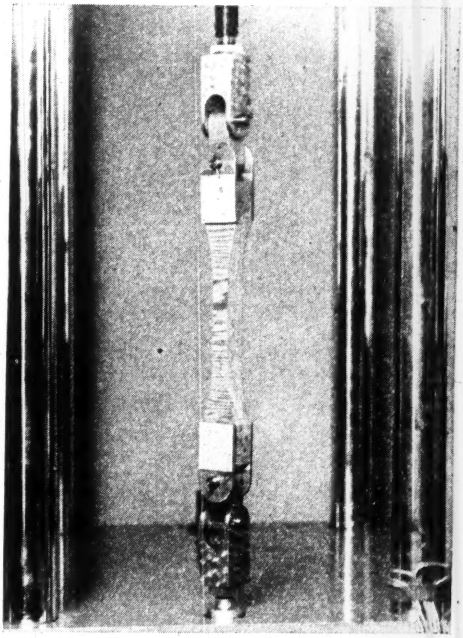

FIG. 10.—Tension-Perpendicular-to-the-Grain Specimen in 4000-lb. Direct-Stress Fatigue Machine, Showing Self-Aligning Pin-Connected Fittings and Steel Plates Which Transmit the Load from the Loading Screws to the Specimen.

Figures 9, 10, and 11, respectively, show tension-parallel-to-grain, tension-perpendicular-to-grain, and laminated glue-shear specimens in the direct-stress fatigue machine. These figures show the plates glued to the specimens, and show the pin-connected fittings that transmit loads from the machine to the plates.

Test Procedure.—One of the direct-stress fatigue machines is shown in Fig. 12. In this machine two specimens can be tested simultaneously, each to a maximum load of 4000 lb. Cross fittings may be used when testing a single specimen to a maximum load of 8000 lb. Both "sides" of the machine are driven from one shaft to which the adjustable eccentrics are attached. The V-belt connects the cam shaft to the motor. With the four sizes of pulleys furnished, loads can be applied to the specimens at rates of 700, 900, 1050, or 1200 cycles per minute with the motor operating at 1200 rpm. Two automatic counting devices, one for each side, are provided for determining the number of cycles to which the specimens are subjected. The setting of the eccentric is dependent upon the variation in load desired. The mean load (preload) is applied to the specimen by means of the upper loading screw, and the desired variation for each cycle is applied through the reciprocating loading arm which is attached to the adjustable eccentric by the vertical connecting rod.

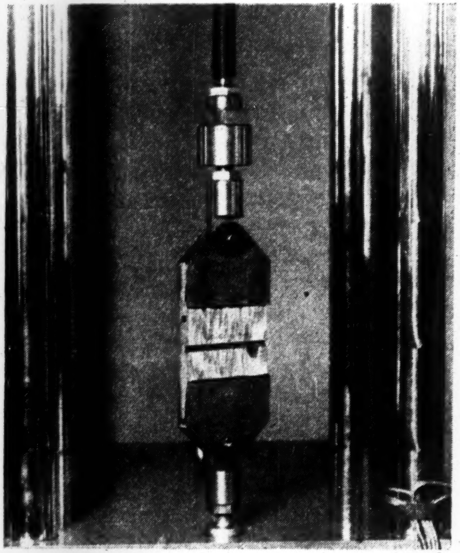

FIG. 11.—Laminated Glue-Shear Specimen in 4000-lb. Direct Stress Fatigue Machine Showing Pin-Connected Fittings Which Transmit the Load from the Loading Screws to the Specimen.

The other direct-stress fatigue machine is similar to the 4000-lb. machine, except that two specimens can be tested simultaneously to a maximum load of 10,000 lb. each. Because it is larger and has greater mass, it is operated at rates of 500, 700, or 900 cycles per minute.

The static loads on the specimens are determined from readings of the load-indicating dial bar (Fig. 12, *F*). The dial measures the deflection of the cantilever loading arm, which deflection is directly proportional to the load. (This dial bar is removed when the machine is running.) Due to inertia effects in the arm and other moving parts, however, the difference in dynamic load from mean to maximum and from mean to minimum is greater than the indicated static differences. This difference was found by using the electric strain gages (Fig. 12, *D*) mounted on the vertical flexure plates, together with an alternating-current, Wheatstone-bridge, cathode-ray oscillograph unit similar to the one described by H. J. Grover (**5**). The ratio of the dynamic difference between maximum and minimum loads to the corresponding static difference was found to be a constant for each machine for each machine speed. This correction factor for the inertia effects has been called the "dynamic throw factor," and corrections are made to the static-dial-bar readings so that the dynamic loads when the machines are operating are the desired loads. With operation at 900 cycles per minute, the dynamic throw factor for the 4000-lb. machine was found to be 1.075 and for the 10,000-lb. machine, 1.120.

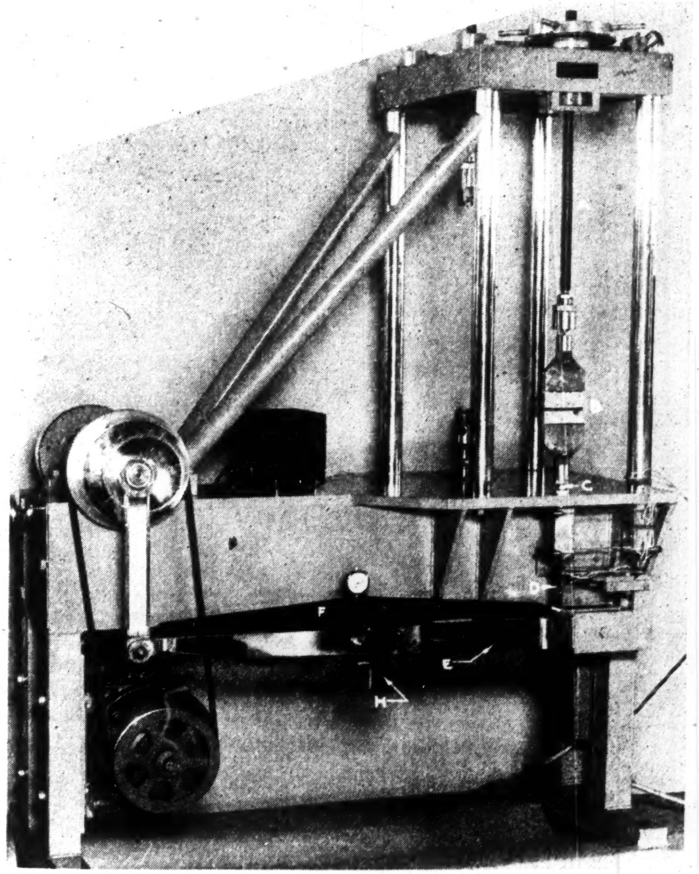

FIG. 12.—Direct-Stress Fatigue Machine.
A—Upper loading screw
B—Specimen
C—Lower loading screw
D—Electric strain gages for determining corrections for dynamic effects
E—Horizontal loading arm
F—Calibrated dial bar for determining static loads
G—Adjustable eccentric
H—Electronic cut-off switch for stopping machine

Each side of the fatigue machines is equipped with an electronic shut-off mechanism (Fig. 12, *H*), which turns off the motor when, because of yield of the specimen, the load decreases more than a predetermined amount. The shut-off mechanism is based on the fact that the deflection of the reciprocating loading arm changes with a change in the amount of load. The amount of reduction in load necessary to operate the relays shutting off the machines is determined by the setting of the cut-off switch, which can be adjusted so a reduction in load of as little as 20 lb. will stop the machine. Thus, it is possible with proper monitoring to achieve essentially constant load.

Because the temperature of a specimen

tends to increase during test, and because wood has a low coefficient of thermal conductivity, fans were installed on the fatigue machines to cool the specimens during test. An increase in temperature during test would tend to lower the moisture content of the specimen and to change its strength, and the adopted technique reduces the effects of this variable. An appreciable rise in temperature in test is undesirable also because it is unlikely to occur in service.

The procedure in making a test is as follows:

1. The adjustable eccentric is set at zero and the reading of the dial bar at zero load determined.

2. The specimen (Fig. 12, *B*) is fastened in the machine, and the clamping nuts are tightened to prevent rotation.

3. The desired mean load is placed on the specimen by operating the upper loading screw (Fig. 12, *A*). The desired deflection reading for the indicating dial bar is computed by multiplying the mean load by the calibrating factor for the dial bar and adding it to the initial reading.

4. The desired variation in static load either side of the mean is placed on the specimen by proper adjustment of the eccentric. This is a cut-and-try procedure, but with a little experience the adjustment is made in three or four attempts. The desired difference of the dial-bar readings is computed by multiplying the difference between either the mean and maximum or the mean and minimum loads (which differences are identical) by the calibrating factor for the dial bar and dividing by the dynamic throw factor.

5. The cycle counter is set at zero and the machine is started.

6. The cut-off switch is next set so that the machine will stop if the load changes more than the desired amount.

7. After approximately 1000 cycles of stress, the machine is stopped, the maximum and minimum loads are checked, and the machine is adjusted if necessary.

8. The machine is again started and monitored when necessary until either

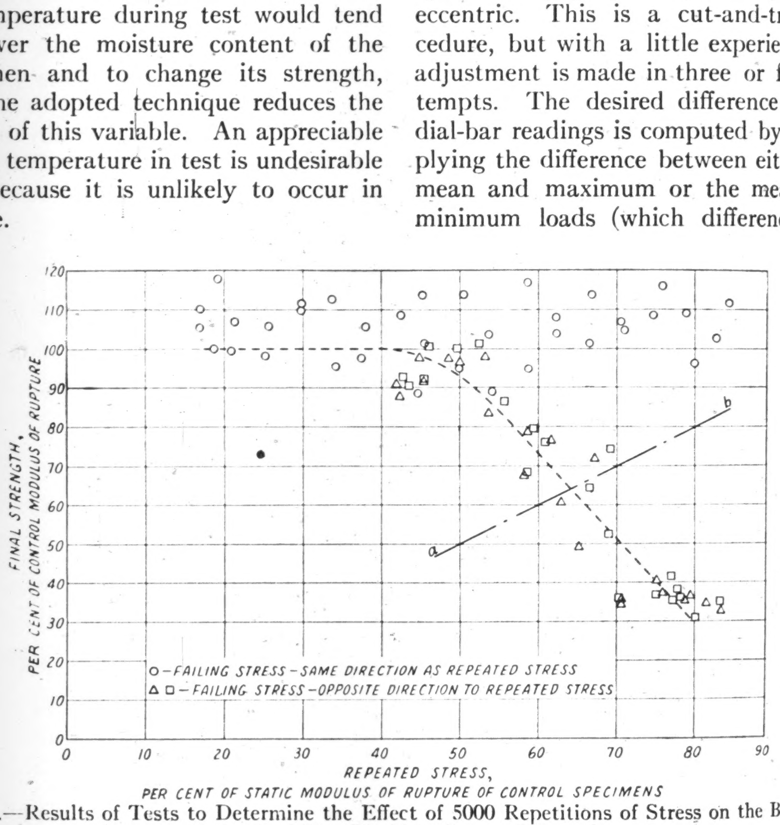

FIG. 13.—Results of Tests to Determine the Effect of 5000 Repetitions of Stress on the Breaking Strength of Five-Ply Sitka Spruce Plywood.

the specimen fails, or 30,000,000 cycles of loading have been completed.

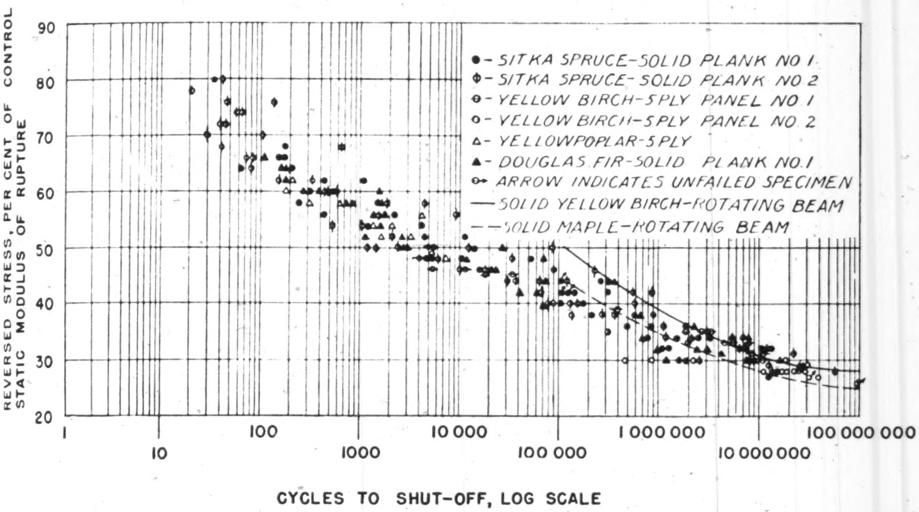

FIG. 14.—Results of Tests to Determine Endurance of Wood and Plywood When Subjected to Reversed Bending Stress. Rotating-beam fatigue data were obtained from results of tests by F. B. Fuller and T. T. Oberg at Wright Field.

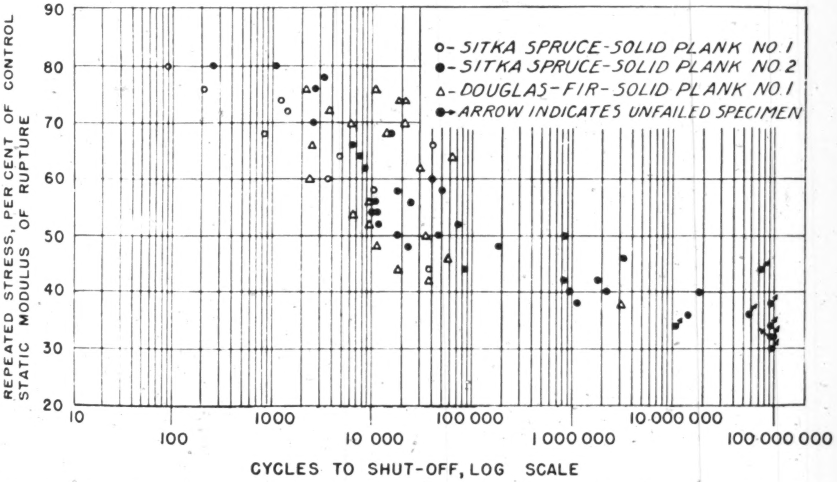

FIG. 15.—Results of Tests to Determine Endurance Strength of Sitka Spruce and Douglas Fir When Subjected to Repeated Bending Stress.

RESULTS OF FLAT-PLATE BENDING TESTS

Effect of 5000 Cycles of Repeated Stress on Ultimate Strength:

Tests were made, as reported by W. J. Kommers (6), on specimens of five-ply Sitka spruce plywood comprised of veneers $\frac{1}{16}$ in. thick of aircraft quality. These specimens were loaded repeatedly 5000 times from zero stress to a maximum value, and immediately tested to destruction under static loading in the same or reversed direction. The results are shown in Fig. 13, where the maximum repeated stresses are plotted as abscissas with the final strengths as ordinates. From this graph, it can be seen that 5000 repetitions of stress to as much as 85 per cent of the ultimate static strength of the controls did not affect the strength adversely for loading in the same direction as the repeated loadings. This graph also seems to suggest that the final breaking strength was increased by the repeated loading. Some of the apparent increase may be due to a lowering of the moisture content of the specimens at the point of maximum stress by the drying effect of the heat evolved (these specimens were not cooled by a fan during test). Specimens tested to failure in the opposite direction, after repetitions of stress exceeding 30 or 40 per cent of the control strength had reduced final strengths. This was undoubtedly due to the effect of compression (due to the repeated loading) on the tensile strength of the extreme fibers. Compression failures were plainly visible in some specimens. Points above the line *a–b* had final breaking strengths which exceeded the value of the repeated stress, and the points below final breaking strengths which were lower than the value of the repeated stress.

Repeated and Reversed Stresses:

A comprehensive series of tests (**4**) was made on specimens of Sitka spruce and Douglas fir wood, and on five-ply yellow birch and yellow-poplar plywoods. The total number of tests, including controls, was approximately 1100. Part of the specimens were subjected to completely reversed stress (mean stress zero) and part to stresses repeated from zero to certain percentages of the modulus of rupture. The results of these reversed bending tests are presented in Fig. 14. The two curves representing rotating-beam tests made by F. B. Fuller and T. T. Oberg at Wright Field are included to show that the end point of test selected (discussed in section on "Methods of Test Developed") is reasonable. The endurance load for 50,000,000 cycles of reversed stress is shown to be approximately 27 per cent of the static modulus of rupture for the species and constructions investigated.

The results of the repeated-bending tests are shown in Fig. 15. The data are not conclusive but suggest, for the Sitka spruce and Douglas fir tested, an endurance load for 50,000,000 cycles of approximately 35 to 40 per cent of the static modulus of rupture. It may be noted from Figs. 14 and 15 that the results of fatigue tests plotted form a band rather than a closely-defined pattern for an S-N curve. The spread is largely due to the variability of the strength of the material itself, rather than to any inconsistencies in the methods of test. It is not possible to determine exactly the static strengths of the fatigue specimens. The practice of closely matching the fatigue specimens to control specimens tends to reduce but does not eliminate this spread.

Tests in repeated and reversed bending indicated that the endurance limit (the maximum stress that can be repeated indefinitely without failure) does not occur at least before 50,000,000 cycles. The S-N curves tend to become asymptotic to a horizontal line and are still a decreasing function at this limit. For ferrous metals, except for very high-strength steels, a life of 10,000,000 cycles affords reasonable assurance that the endurance limit has been reached. Thus it appears that when evaluating the fatigue characteristics of wood and glued-wood constructions, the results should be discussed in terms of endurance loads for some certain number of cycles of loading. Fifty million cycles have been chosen for the flat-plate bending tests.

Reversed Stress Superimposed on Steady Stress:

Tests were made on specimens of solid Sitka spruce and Douglas fir (7) with a reversed loading cycle superimposed on a steady stress to simulate the action of gust loads on an airplane in nonaccelerated flight. In one series, the mean stress was constant at 20 per cent of the modulus of rupture, and the reversed stress cycles covered a range from 15 to 60 per cent of the modulus of rupture, thus giving maximum stresses that varied from 35 to 80 per cent of the static modulus of rupture. In a second series, the maximum stress was held constant (60 per cent of the static modulus of rupture) and the midpoint of the deflection cycle varied to give mean stresses equal to different percentages (0 to 30 per cent) of the modulus of rupture.

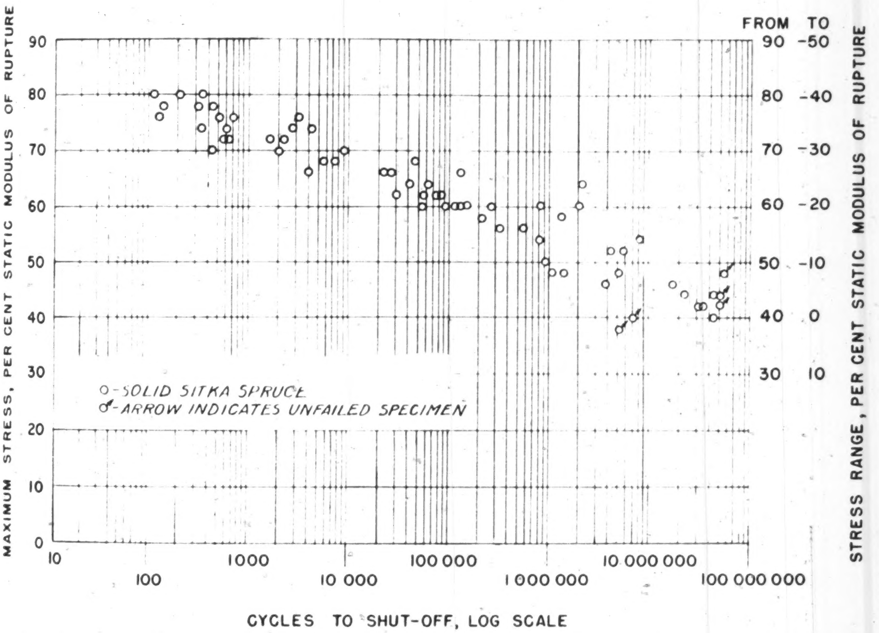

FIG. 16.—Results of Tests on Sitka Spruce with the Mean Stress Held at 20 per cent of the Modulus of Rupture and the Maximum Stress Varied from 80 per cent down to 38 per cent of the Modulus of Rupture.

The first series included Douglas fir and Sitka spruce, and the second was on Sitka spruce only.

The data for 20 per cent mean stress for Sitka spruce are plotted in Fig. 16. (A similar plot was obtained for the tests of Douglas fir.) This plot shows the endurance load for 50,000,000 cycles as a reversed stress of about 19 per cent of the modulus of rupture superimposed on the mean. This corresponds approximately to a repeated stress cycle from zero to 40 per cent and is in reasonable agreement with Fig. 15. Similar results were indicated by the tests on Douglas fir.

Figure 17 shows the results of a limited number of tests of Sitka spruce subjected to stress cycles having a maximum stress of 60 per cent of the static modulus of rupture with various values of the mean stress. When the mean stress was zero, the stress cycle was completely reversed from +60 to −60 per cent. When the mean stress was 30 per cent, the stress cycle was of

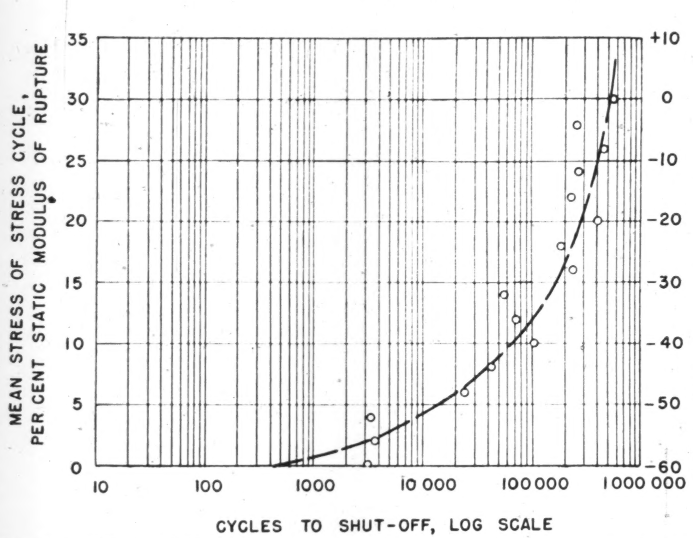

Fig. 17.—Results of Tests of Sitka Spruce Specimens Subjected to Stress Cycles Having the Same Maximum Stress, 60 per cent of the Modulus of Rupture, but with Varying Mean Stresses.

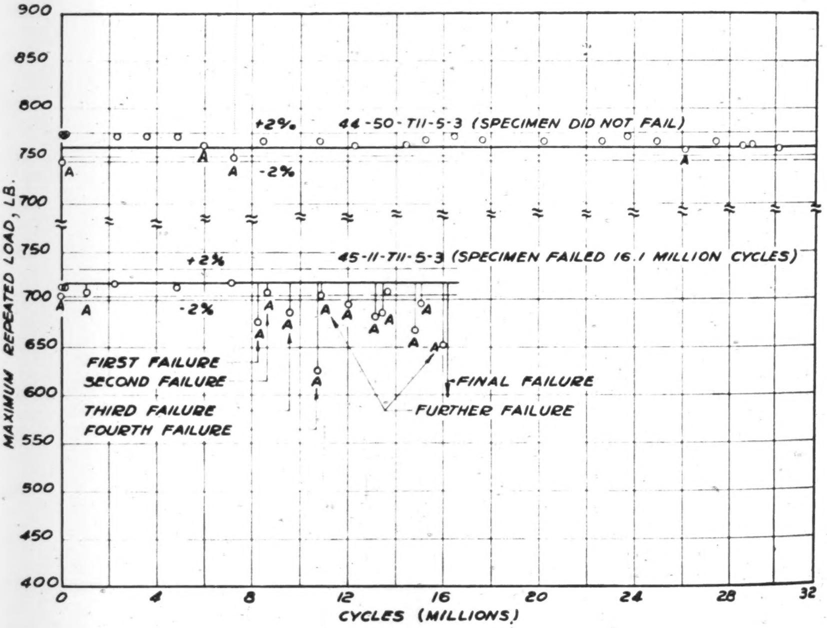

Fig. 18.—Typical Variation of Maximum Repeated Load with Cycles of Stress for Tension-Parallel-to-Grain Test of Douglas Fir.

the repeated type from zero to a maximum of +60 per cent. At each of these points, the indicated endurance life is considerably greater than is indicated by Figs. 14 or 15.

Results of Direct-Stress Fatigue Tests

Tension Parallel to Grain:

Direct-stress fatigue tests in tension parallel to the grain are being completed

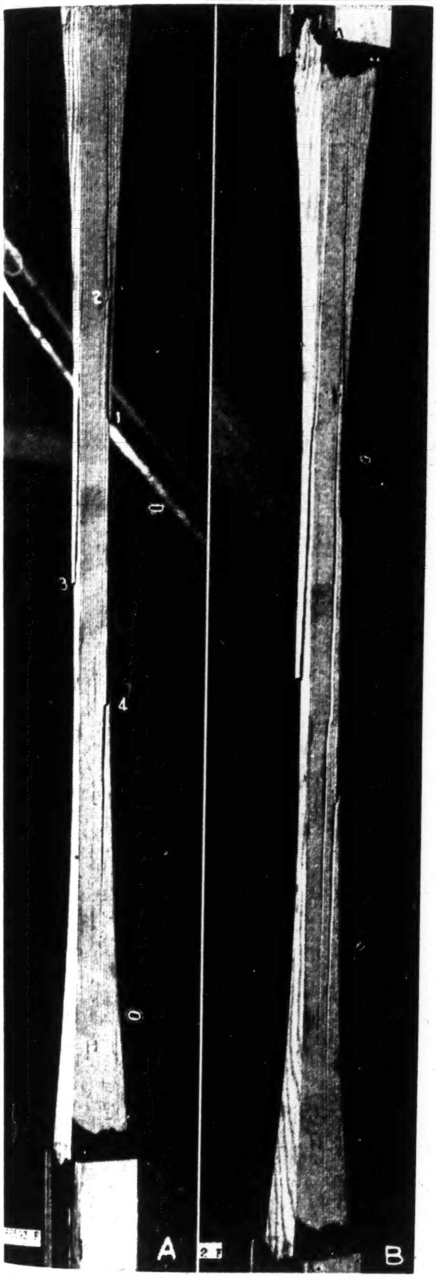

FIG. 19.—Typical Fatigue Failure of Douglas Fir in Tension Parallel to Grain, Showing Extent of Failure at 11,000,000 Cycles (*A*), and at 14,000,000 Cycles (*B*). Numbers refer to sequence of failures.

on specimens of Douglas fir and white oak. For several years, and particularly during the war, considerable use has been made of laminated construction in which structural members are formed by gluing boards together; often it is necessary to join boards end to end by means of scarf joints or joints of other types to make laminations of the required length. This series of tests was undertaken to determine how scarf-jointed specimens compared with solid specimens when subjected to fatigue loadings and to obtain fundamental information on fatigue of wood in tension. The slope of scarf used was 1:8, and it was recognized that this joint could not be so strong as a specimen without a joint. The fatigue specimens of solid wood were loaded so that the maximum repeated loads were specific percentages of the strengths for solid control specimens, and the scarf-jointed fatigue specimens were loaded to similar percentages of the strengths of the scarf-jointed control specimens. The ratio of the minimum to the maximum repeated load (range ratio) was 0.10 for all tests.

Figure 18 presents plots of the maximum repeated loads as found from periodic measurements *versus* the number of cycles for two typical Douglas fir tension specimens. The upper graph applies to a specimen that was subjected to 30,000,000 repetitions of 50 per cent of the static ultimate strength without failure and the lower to a specimen also subjected to 50 per cent of the static ultimate strength, which developed the first failure at over 8,000,000 cycles and had further failures until at 16,000,000 cycles it was no longer capable of sustaining the required load. Each failure stopped the testing machine; adjustments were made to bring the load back to the desired amount before continuing the test. The letter *A* indicates that either the maximum or minimum loads, or both, were adjusted at this point in the test. The solid lines show the desired maximum repeated loads, and the dotted lines are reference lines 2 per cent above and below the desired maximum repeated loads. These plots show that practically constant loads can be maintained with this machine, which applies essentially constant deformations.

Failures during two stages of the test of the second specimen, discussed in the preceding paragraph, are presented in Fig. 19. The numbers refer to the sequence of failure. The illustration at the left shows the extent of failure at 11,000,000 cycles and that at the right the extent at 14,000,000 cycles. These failures are typical fatigue failures in tension for Douglas fir and are identical in appearance to the static failures. Douglas fir, having a comparatively long fiber, usually has a long splintering type of failure in tension unless it has a low specific gravity or some defect is present. White oak has shorter fibers and the specimens tested had more brashy or square-appearing failures in tension parallel to the grain, but again no difference between the appearances of the fatigue and the static failures could be discerned. It is possible that fatigue failures in wood escape detection as such because of their similarity to ordinary structural failures.

The results of these tests for both

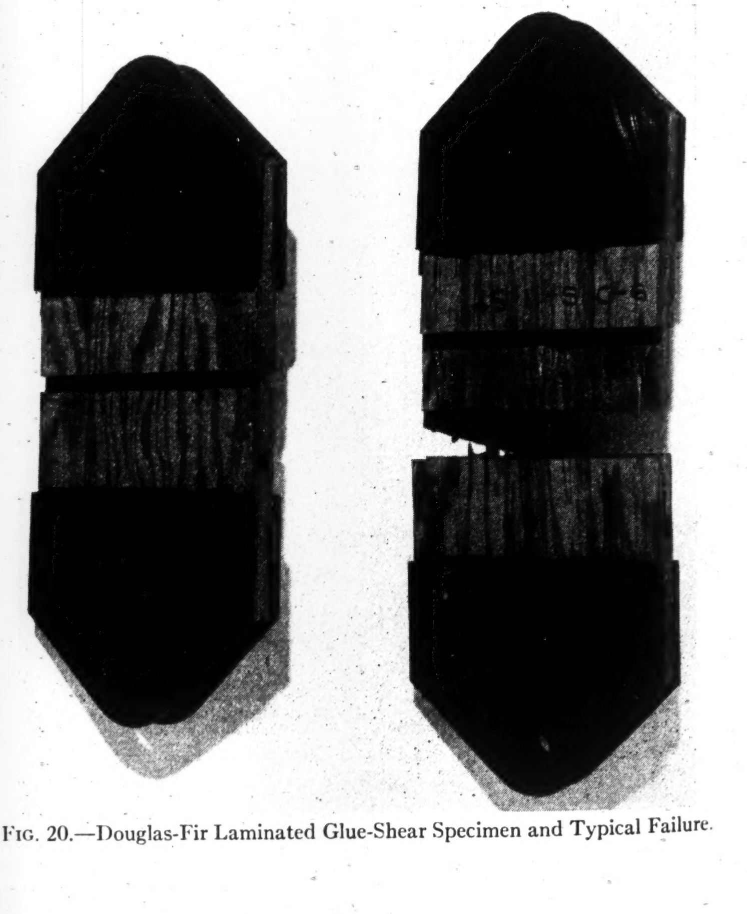

FIG. 20.—Douglas-Fir Laminated Glue-Shear Specimen and Typical Failure.

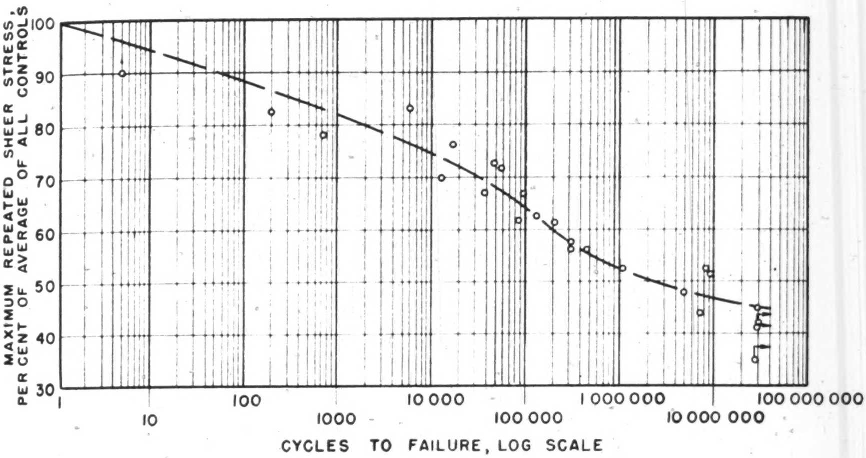

FIG. 21.—Results of Tests of Laminated Glue-Shear Specimens of Douglas Fir. The ratio of minimum to maximum loads (range ratio) was 0.10 for all tests.

species, though all analysis is not complete, indicate that scarf-jointed specimens behave as well under repeated loadings as solid specimens. The average endurance load for 10,000,000 cycles for both types of specimens of Douglas fir is approximately 50 per cent of the static ultimate strength, when the range ratio is 0.10. The failures in fatigue of the scarf-jointed specimens also have the same appearance as the static failures.

Laminated Glue Shear:

The tests of the glued laminated specimens in which the joints are stressed in shear are complete for one species, Douglas fir. A cold-setting resorcinol resin glue was used in fabricating these specimens, and again the fatigue failures have exactly the same appearance as the failures due to static loadings. Figure 20 shows a typical failure of the laminated shear specimen. It may be noted that there was no evidence of deterioration of the glue and that the failure was almost entirely a shear failure of the wood.

Figure 21 presents the *S-N* curve obtained from the tests of Douglas fir in glue-shear. It is a semilogarithmic plot with the maximum repeated loads expressed as percentages of the average static shear strength plotted to a uniform scale as ordinates and the cycles to failure plotted to a logarithmic scale as abscissas. The range ratio for these tests was constant (0.10), the same as for the tension-parallel-to-grain tests. The average static strength of the specimens was 750 psi. From this curve, the endurance load for 30,000,000 cycles is estimated as approximately 45 per cent of the static strength. This curve is similar to the curves for the cantilever-bending fatigue tests in that it does not indicate a definite endurance limit before 30,000,000 cycles and further illustrates that the results of fatigue tests of wood and glued-wood constructions should be reviewed in terms of endurance loads for a specified number of cycles rather than for any estimated endurance limit.

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(3) C. B. Smith, "The Effect of Elliptic or Circular Holes on the Stress Distribution in Plates of Wood or Plywood Considered as Orthotropic Materials," Report No. 1510, issued by Forest Products Laboratory in cooperation with the Army-Navy-Civil Committee on Aircraft Design Criteria, May, 1944.

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(5) H. J. Grover, "The Use of Electric Strain Gages to Measure Repeated Stresses," *Experimental Stress Analysis*, Vol. 1, No. 1, 1943.

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(7) W. J. Kommers, "The Fatigue Behavior of Douglas-fir and Sitka Spruce Subjected to Reversed Stresses Superimposed on Steady Stresses," Report No. 1327-A, issued by the Forest Products Laboratory in cooperation with the Army-Navy-Civil Committee on Aircraft Design Criteria, May, 1944.

DISCUSSION

Mr. L. R. Jackson.[1]—I should like to ask whether, in the study of fundamentals of wood, any work has been done on speed effect. Perhaps the speed at which these tests are run might make some difference.

Mr. Wayne C. Lewis (*author*).—The effect of speed of repetition of stresses in the fatigue of wood is probably appreciable, but no specific study of this phenomenon has been made to date. We are trying to get as much of the survey type of information to the industry as possible. The refinements will come later.

The strength properties of wood are affected by changes in the moisture content. The work absorbed by the specimen during the repeated loading is dissipated mainly as heat. Wood has a low coefficient of thermal conductivity so that the rise of temperature is considerable if the rate of repetitions of load is high. This increase of temperature is noticeable at 900 cycles per minute at the higher levels of stress. Any increase in temperature in the specimen would tend to decrease the moisture content, thereby increasing the strength.

I neglected to state in my presentation that we have provided fans on the direct stress machines to cool the specimens under test and thus keep the temperature rise to a minimum.

In Fig. 13, it appeared that the strengths of the Sitka spruce plywood were greater after 5000 repetitions of stress than before. Small coupons were cut from the specimens at the point of failure, and from the moisture-content determinations it was found that the moisture content was lower at the points of maximum stress. This probably accounts for the apparent increase in strength and illustrates that the speed effect would be considerable for wood.

The idea of cooling the specimens under test appears to be a good one for tests of wood, where the heat generated would tend to dry out the specimens, or for tests of plastics where an increase of temperature would change the elastic properties of the material.

Mr. F. R. Shanley.[2]—I should like to know whether the failure you referred to is actually a shear failure in some cases. It may have been a cross-grain failure, particularly for the specimen with the hole.

If the so-called shear failure progressed through the center of the hole, and the hole was in the middle of the specimen, it is difficult to see how there could have been a shear stress there.

This is of special interest in connection with metals, because, as you know, there are certain types of metals which have much lower properties across the grain than with the grain.

Mr. Lewis.—When the primary stress is in shear it is often difficult to determine the fundamental mode of failure. Actually, what is apparently a shear failure may be a combination of shear and tension perpendicular to grain. A shear stress can be resolved into diagonal compression and tension, and if the material is relatively weaker in these directions it will be reflected in the mode of failure.

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[1] Supervisor, Battelle Memorial Institute, Columbus, Ohio.

[2] Lockheed Aircraft Corp., Burbank, Calif.

In the case of the hole in the tension flange of the box spar, the shear failures were approximately tangent to the hole, started at the hole and progressed longitudinally as the test continued. If the grain were straight and parallel to the length of the flange, we had a slot the diameter of the hole wide throughout the critical section of the beam at the final failure in tension parallel to the grain.

This is discussed in detail in Forest Products Laboratory Report No. 1510, "The Effect of Elliptic or Circular Holes on the Stress Distribution in Plates of Wood or Plywood Considered as Orthotropic Materials," by C. B. Smith, and in Report No. 1513, "Effects of Certain Defects and Stress-Concentrating Factors on the Strength of Tension Flanges of Box Beams," by W. C. Lewis, T. B. Heebink, and W. S. Cottingham.

MR. JOHN M. LESSELLS.[3]—In the case of the failure of metals considerable light has been thrown on the mechanism of failure by the combined efforts of the metallurgist and engineer. The users of the microscope and X-ray have added considerably to our knowledge on the mechanism of failure of metals.

In the case of wood the problem is more complex and it would be interesting to know to what extent these tools have been used for analyzing the failure of wood.

A further point is that in the case of steel and concrete we are dealing with artificial products whereas wood is a natural product. The question is whether we are going to improve this product of nature either during growth or by impregnation with plastics at a later stage.

CHAIRMAN R. E. PETERSON.[4]—I think Mr. Lessells has raised a very interesting question, involving fundamentals of failure.

I think most of you could not help but notice, from one of the figures, that the shape of the fatigue curve for wood was almost the same as you might get for aluminum alloys, or many other metals, as far as that is concerned.

We have seen many theories as to what governs the shape of these curves, these theories being made by people who work with metals, involving grain size, cold working and so on. We must realize that such theories will not apply to all materials, and here is a material which must have a quite different mechanism of failure.

MR. LEWIS.—Many theories have been advanced on the development of fatigue failures and several good techniques have evolved for determining the extent of damage due to fatigue loading of metals. How these techniques may be used in the study of fatigue of wood is not known. The press of other work has prevented any study of the mechanics of the fatigue failures of wood.

Because thin sections of wood are translucent and light will pass through them, it may be possible to get considerable information on the phenomenon of failure from microscopical studies. For example, some species of wood sometimes have minute compression failures put in them during growth by snow or wind loads, or at the time of felling by falling against or across another tree. These pre-existing compression failures are detected easily when viewed under a microscope with polarized light.

MR. J. O. ALMEN.[5]—I should like to know the extent of the variability which exists under static loading. What would be the amount of scatter if the fatigue tests were carried back to a small number of stress cycles—for example, ten to one hundred cycles? We have run a large number of fatigue tests on cast iron, and we find the variability to be great—something on the order of ten or twenty to one. In a tension test on cast iron, the scatter would be relatively small.

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[3] Associate Professor of Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

[4] Manager, Mechanics Div., Westinghouse Research Labs., Westinghouse Electric Corp., East Pittsburgh, Pa.

[5] Head, Mechanical Engineering Dept., Research Labs. Div., General Motors Corp., Detroit, Mich.

In the case of wood, is the scatter greater at a single load application than under repeated loadings? For all metals used in machine structures, the scatter under repeated loadings is far greater than under static loading. Since all these materials have some ductility, local high stresses are adjusted by yield so as to avoid the notch effects that occur under conditions of fatigue. Therefore, the variation in the static strength is small.

MR. LEWIS.—Because wood is a natural material that is formed during many years of growth, many things are likely to affect its strength and considerable variation in results of tests can be expected, regardless of whether the specimens were failed by a single application of load or many. This variation is greater for some strength properties than others.

It can be expected that the spread of values would be greater for the fatigue tests than for static tests because, in general, points of stress concentration affect fatigue values more than static values. Such stress raisers are prevalent in a natural material.

If 15 specimens were tested in static bending from a single plank of wood, a coefficient of variation for the values of modulus of rupture could be expected to be about 10 per cent. The individual values might vary considerably but the mean value obtained would give a reasonable measure of the strength property for the plank.

MR. JACQUES L. BIENFAIT.[6]—When I was working at the Forest Products Laboratory in 1923 and 1924, I found myself caught in the lure of trying to find out something about the relation of timber failure to the structure of the timber.

As I was working there only about 10 months, I had the opportunity then to complete only a study[7] in a certain limited number of timbers. However, I can assure you that this lure, of trying to know the why and the how of failure, in that particular field of timber, has never left me.

Fortunately in your own country there are many people who work in this particular field, in universities and laboratories, and it is also being continued at the Forest Products Laboratory.

Of course, the field of commercial timber is so large that the problem necessarily has to be limited, and the time that any one man can put in, in this field, is also a limitation to obtaining a solution. However, we have with timber the possibility of looking at specimens and slides, with the help of microscopic slides and the polarized light microscope.

I do think progress will be made along those lines in the future, when the efforts of the world scientists are combined.

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[6] Partner, Messrs. Koning and Bienfait, Materials Testing Labs., Amsterdam, Holland.

[7] Jacques L. Bienfait, "Relation of the Manner of Failure to the Structure of Wood Under Compression Parallel to the Grain," *Journal of Agricultural Research*, Vol. 33, No. 2 (1926).

INVESTIGATION OF MATERIALS FOR MARINE PROPELLERS*[1]

By William C. Stewart[2] and W. Lee Williams[2]

Synopsis

This paper presents the results of tests on 32 materials, mostly cast, which have been used or proposed for use in marine propellers. The tests have included determinations of the chemical compositions and mechanical properties. In addition, the alloys have been examined from the standpoint of resistance to salt water corrosion, salt water cavitation, and the effects of simultaneous cyclic stress and salt water corrosion.

During the past few years renewed interest has been shown in the study of materials for marine propellers. Several investigations have been conducted to review critically some of the older, well-known alloys and to compare them with new compositions which have been proposed from time to time (**1, 2**).[3] In the production of high-speed vessels, a trend toward the use of thinner blade sections and higher blade velocities has stimulated considerably the search for better propeller alloys. The trend has created a condition in which greater emphasis has been placed on such properties as high strength, resistance to the combined effects of cyclic stress and salt water corrosion, and resistance to cavitation erosion (**2, 3, 4, 5**). The enhancement of these properties has had to be accomplished without too much sacrifice of the ease and cost of propeller production, fabrication, and maintenance.

This paper presents the results of a rather comprehensive study on the chemical and mechanical properties of a number of old and proposed propeller alloys. Whereas no attempt has been made to include all the materials which might be suitable, the alloys which were studied do represent a fairly good cross-section of the most promising ones available for the purpose intended.

Materials Investigated

In Table I are listed the compositions of the 32 materials included in the investigation. As nearly as possible, the alloys are listed systematically according to their compositions. In certain instances the material designations will be used for convenience in the identification of particular materials.

It may be noted that five of the alloys are manganese bronzes similar to, or meeting, Navy Specification 49B3. These five alloys represent a type of material that has been used in marine propellers for many years and thereby provide an excellent basis for comparison with the other compositions.

With the few exceptions noted in the

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] The views presented in this paper are those of the authors and do not represent necessarily the opinions of the Navy Department.

[2] Principal Metallurgist and Metallurgist, respectively, Engineering Experiment Station, Annapolis, Md.

[3] The boldface numbers in parentheses refer to the references appended to this paper, see p. 845.

table, each material was obtained in the form of castings measuring about 12 in. square and 2 to 2½ in. thick. A section this size was considered to be approximately representative of an actual propeller. It is of interest to note that manganese bronze material CHC was removed from a 9½-in. thick section near the hub of a large marine propeller. The properties obtained in such a large casting can be compared with the smaller sections of the other manganese bronzes.

All the materials were considered to be reasonably sound castings. Certain other alloys, originally intended for inclusion in the investigation, were eliminated because of excessive porosity. Except as noted in the table, the various alloys were tested in the as-cast condition.

TABLE I.—MATERIAL UNDER TEST.

Unless specified below, each material was cast in plates 12 in. square and 2 to 2½ in. thick.
Except as specified below, materials were tested in the as-cast condition.

Material Designation	Type of Material	Chemical Composition, per cent															
		C	S	P	Mn	Si	Cr	Ni	Cu	Zn	Pb	Al	Sn	Fe	Be	Mg	C
OV	16-6 Corrosion resistant steel	0.05	0.013	0.019	0.48	1.46	16.54	6.00	....	....	....	....	....	....	....	....	....
PC	20-10 Corrosion resistant steel[a]	0.05	0.015	0.040	0.52	0.24	20.50	10.20	0.14	....	....	....	....	....	....	....	....
OW	22-11 Corrosion resistant steel	0.05	0.009	0.033	0.52	1.68	22.20	10.88	....	....	....	....	....	....	....	....	....
RG	Corrosion resistant iron[b]	0.09	0.026	0.018	0.72	0.96	12.57	....	....	....	....	....	....	....	....	....	....
QQA	Cu-Zn-Sn alloy	....	....	....	....	....	....	....	83.60	14.37	....	0.08	1.64	0.36	....	....	....
QQC	Cu-Zn-Sn alloy	....	....	....	....	....	....	....	85.70	6.22		0.19	7.58	0.33	....	....	....
CHC	Mn bronze[c]	....	....	....	0.78	....	....	....	57.93	39.18	Nil	0.65	0.78	0.69	....	....	....
OX	Mn bronze (min. alpha)	....	....	....	0.30	....	....	....	56.52	41.26	Nil	1.11	Nil	1.13	....	....	....
OY	Mn bronze (virgin metal)	....	....	....	0.29	....	....	....	57.12	41.00	Nil	0.62	Nil	1.10	....	....	....
OZ	Mn bronze (beta phase)	....	....	....	1.14	....	....	....	53.20	42.79	Nil	1.22	Nil	1.75	....	....	....
PA	Mn bronze (50 per cent scrap metal)	....	....	....	0.75	....	....	....	58.30	38.62	Nil	0.93	0.46	1.20	....	....	....
PB	High tensile Al-Mn bronze	....	....	....	3.86	....	....	....	65.30	20.24	Nil	7.81	Nil	2.79	....	....	....
RI	High tensile Al-Mn bronze	....	....	....	4.73	0.02	....	....	66.69	21.82	....	4.29	....	2.45	....	....	....
QU	Mn-Ni bronze	....	....	....	2.86	....	....	1.72	48.99	44.90	....	....	0.41	1.12	....	....	....
OUA	Al-Ni bronze[d]	....	....	....	Nil	0.96	....	3.36	89.90	....	....	4.86	....	0.42	....	....	....
OUB	Al-Ni bronze	....	....	....	0.02	0.88	....	3.40	90.66	....	....	4.40	....	0.11	....	....	....
ADU	Al-Ni bronze	....	....	0.039	0.71	0.15	....	12.70	84.19	....	....	1.16	....	1.05	....	....	....
RD	Al-Ni-Fe bronze	....	....	....	....	....	....	5.03	79.75	....	....	10.07	....	5.09	....	....	....
PJ	Al bronze	....	....	....	....	....	....	....	86.42	....	....	10.20	....	3.37	....	....	....
OT	Si bronze[e]	....	....	....	0.11	4.23	....	....	93.58	Nil	....	....	....	2.46	....	....	....
PK	Ni-Cu alloy	0.18	....	....	0.74	0.06	....	62.72	34.76	....	....	0.70	....	0.86	....	....	....
PS	Ni-Cu alloy[f]	0.31	....	....	0.84	1.10	....	65.67	29.57	....	....	0.93	....	1.58	....	....	....
PR	Ni-Cu-Si alloy[f]	0.25	....	....	0.48	2.88	....	63.00	29.65	....	....	1.48	....	2.26	....	....	....
RE	Ni-Cu-Si alloy	....	....	....	0.62	3.65	....	66.14	28.10	....	....	....	....	1.49	....	....	....
PO	Ni-Cr alloy[g]	0.05	....	....	1.07	0.20	18.04	73.28	2.10	....	....	2.14	....	3.19	....	....	....
QQ	Cu-Be alloy[h]	....	....	0.025	....	0.12	....	....	97.04	....	....	....	....	0.10	2.91	0.33	....
QJC	Cu-Ni-Be alloy[i]	....	....	....	....	....	....	1.64	97.91	....	....	....	....	0.13	0.31	....	....
QY	Cu-Ni-Be alloy[j]	....	....	....	....	....	....	1.99	95.65	....	....	....	....	0.19	2.19	....	....
QJA	Cu-Ni-Be alloy[k]	....	....	....	....	....	....	2.48	95.09	....	....	....	....	0.02	2.36	....	....
QJD	Cu-Co-Be alloy[l]	....	....	....	....	....	....	....	96.46	....	....	....	....	0.32	0.53	0.16	2.85
AAD	Mn-Cu-Ni alloy	0.09	....	....	72.59	....	....	9.54	17.78	....	....	Nil	....	Nil	....	....	....
AAE	Mn-Cu alloy	0.04	....	....	81.51	0.06	....	....	18.14	....	....	Nil	....	0.25	....	....	....

[a] Forged
[b] 8 by 2½ by 1½ in.
[c] From propeller section, 9½ in. thick.
[d] 2⅝-in. diameter by 24 in. heat-treated.
[e] 12 by 12 by 1 in.
[f] 12 by 6 by 2 in.
[g] 12 by 6 by ½ in. and 1-in. diameter by 12 in.
[h] QQ heat-treated as follows: QQ-525—4 hr. at 525 F.
QQ-575—4 hr. at 575 F.
[i] Heat-treated 2 hr. 400 C.
[j] From propeller; heat-treated 4 hr. 1500 F., quenched. 4 hr. 350 C.
[k] Heat treated 4 hr. 350 C.
[l] Heat-treated 2 hr. 500 C.

METHODS OF TEST

Mechanical Testing:

Before the results of mechanical tests are presented, some of the properties reported will be defined and the conditions of test described. Tension and compression tests were made on a hydraulic machine, the load scale of which was accurate to within 0.10 per cent as determined by a Bureau of Standards certified proving ring. Tension tests were made with standard 0.505-in. specimens. The proportional limit was measured as the maximum stress which could be applied with the strain remaining directly proportional to the stress. This property is a function of the sensitivity of the extensometer; hence, the values reported are for an instrument indicating to 0.0001 in. per inch of the test length. The proof stress is defined as the stress which will produce a permanent set of 0.0002 in. in a gage length of 2 in. The yield strength values were obtained with an automatic stress-strain recorder and represent the stress at 0.5 per cent permanent set.

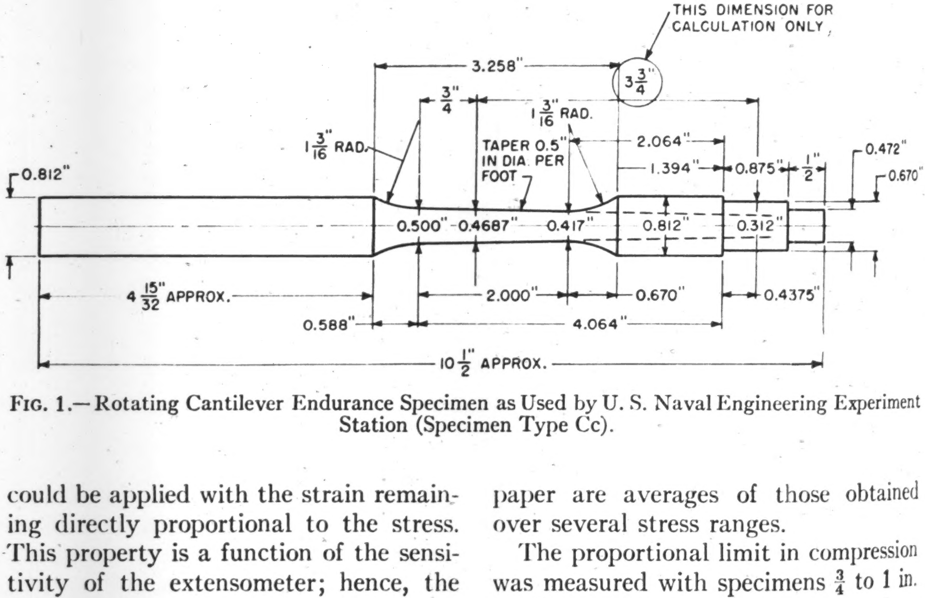

FIG. 1.—Rotating Cantilever Endurance Specimen as Used by U. S. Naval Engineering Experiment Station (Specimen Type Cc).

The modulus of elasticity was determined for the several materials, employing specimens other than those used for the tension tests. The extension was determined by means of two type A Huggenberger extensometers mounted on opposite sides of a standard 0.505-in. specimen. These extensometers were mated, the lever ratio for each being 1200:1. Each graduation of the scale indicated 0.0000208-in. extension per inch of test length. Readings were estimated to one-quarter division. In making these tests it was found that the modulus for some of the materials changed slightly with the stress range. The modulus values reported in this paper are averages of those obtained over several stress ranges.

The proportional limit in compression was measured with specimens $\frac{3}{4}$ to 1 in. in diameter, the length being three times the diameter. The test specimens were mounted in a compressometer provided with two 0.0001-in. dial gages located 180 deg. apart.

Fatigue tests were made with White-Souther rotating cantilever machines, using the specimen shown in Fig. 1. The test length of the specimen is tapered conically and is so designed that the maximum stress occurs $\frac{3}{4}$ in. out from the inner fillet; the stress varies

TABLE II.—RESULTS OF MECHANICAL AND CORROSION TESTS.

Material Designation	Type of Material	Tensile Strength, psi.	Proof Stress, psi.	Yield Strength, psi.	Proportional Limit, psi.		Elongation in 2 in., per cent	Reduction of Area, per cent	Brinell Hardness, 3000 kg. load	Charpy Keyhole Impact Value, ft-lb.	Endurance Limit, psi.	Salt Water Fatigue Tests[b], psi.	Modulus of Elasticity, psi. × 10⁶	Salt Water Corrosion Characteristics (See Text)
					Tension	Compression								
OV........	16-6 corrosion-resistant steel	101 200	27 200	41 600	17 800	18 400	14	22	192	79	26 000	21 000	31.0	Contact pitting
PC........	20-10 corrosion-resistant steel	79 400	24 000	38 400	13 600	17 500	68	75	138	78	30 500	28 000	30.8	Contact pitting
OW........	22-11 corrosion-resistant steel	75 600	21 800	43 400	9 200	18 400	49	74	166	82	22 500	17 500	31.0	Contact pitting
RG........	Corrosion-resistant iron	103 400	67 200	81 600	48 800	......	24	49	220	..	......	......	31.0	Contact pitting
QOA........	Cu-Zn-Sn alloy	27 900	9 800	13 900	7 000	9 600	14	12	65[a]	28	6 700	5 500	18.6	Slight pitting
QOC........	Cu-Zn-Sn alloy	21 800	8 900	14 400	5 500	8 600	11	10	64[a]	13	7 500	6 000	13.1	Slight pitting
CHC........	Mn bronze	55 600	15 000	30 000	......	......	18	16	106[a]	..	10 000	......	......	
OX........	Mn bronze (min. alpha)	78 800	15 600	29 000	10 000	15 600	25	22	150	22	9 000	9 000	15.6	Severe dezincification
OY........	Mn bronze (virgin metal)	73 200	16 400	27 800	9 100	14 200	26	26	147	23	9 000	9 000	15.0	Severe dezincification
OZ........	Mn bronze (beta phase)	85 500	15 800	29 400	8 200	12 100	32	28	160	22	10 200	10 000	14.95	Severe dezincification
PA........	Mn bronze (50 per cent scrap metal)	71 100	14 000	30 600	6 800	8 900	37	33	138	21	13 500	12 500	15.3	Severe dezincification
PB........	High tensile Al-Mn bronze	102 800	34 500	65 800	26 200	23 600	16	16	208	13	17 500	16 500	15.5	Moderate dezincification
RI........	High tensile Al-Mn bronze	110 800	62 500	89 800	46 200	38 200	10	12	232	13	16 000	16 000	16.1	Severe dezincification
QU........	Mn-Ni bronze	87 400	24 400	41 800	20 000	19 800	16	16	176	16	13 500	9 500	15.6	Dezincified pitted lightly
OUA........	Al-Ni bronze	49 800	36 200	46 200	24 400	24 900	4	6	160	16	15 000	......	16.1	Intergranular corrosion
OUB........	Al-Ni bronze	73 400	39 600	50 600	28 000	30 000	11	18	165	16	9 200	8 000	16.3	Intergranular corrosion
ADU........	Al-Ni bronze	67 000	36 000	43 500	39 000	......	11	17	170	..	11 000	8 000	15.8	
RD........	Al-Ni-Fe bronze	88 000	27 600	40 300	22 200	21 600	13	12	172	8	23 000	20 000	20.2	Severe dealuminization
PJ........	Al bronze	85 600	14 200	32 200	7 600	6 800	24	23	160	22	25 000	23 500	17.4	Slight dealuminization
OT........	Si bronze	49 600	13 100	25 000	5 000	9 200	16	15	120	10	15 000	13 000	15.4	
PK........	Ni-Cu alloy	74 000	19 100	27 200	10 000	14 600	48	50	105[a]	39	18 000	12 500	25.2	Good resistance
PS........	Ni-Cu alloy	52 400	15 500	25 000	8 800	13 400	29	31	106[a]	26	12 000	10 000	21.5	Good resistance
PR........	Ni-Cu-Si alloy	92 300	46 800	72 200	34 500	34 600	8	19	230	14	15 000	11 000	25.9	Good resistance
RE........	Ni-Cu-Si alloy	136 500	82 000	102 200	60 000	65 200	10	15	316	25	16 000	13 000	30.2	Good resistance
PO........	Ni-Cr alloy	62 600	31 400	39 500	11 200	19 400	4	7	170	11	18 000	Indef.	27.4	
QQ-525......	Cu-Be alloy	150 000	80 200	....	51 500	......	2	0	375	3	17 500	13 500	19.7	[c]
QQ-575......	Cu-Be alloy	119 600	107 800	....	83 500	97 000	1	0	396	4	15 500	15 500	21.0	[c]
QJC........	Cu-Ni-Be alloy	32 100	25 500	....	......	48 400	1	1	221	13	8 000	7 700	19.8	
QY........	Cu-Ni-Be alloy	114 400	102 500	....	......	......	1	2	388	..	......	......	......	
QJA........	Cu-Ni-Be alloy	107 800	72 500	....	40 000	57 900	2	2	310	6	13 000	12 500	17.2	
QJD........	Cu-Co-Be alloy	49 000	31 200	....	24 000	29 300	2	4	198	16	7 000	7 000	19.95	[d]
AAD........	Mn-Cu-Ni alloy	55 200	10 500	18 900	7 000	......	52	56	82[a]	..	8 000	2 500	15.0	Severe intergranular corrosion
AAE........	Mn-Cu alloy	62 200	6 000	17 800	3 000	......	27	55	135[a]	..	8 000	......	8.2	Severe intergranular corrosion

[a] 1500-kg. load.
[b] See text for significance of corrosion fatigue values.
[c] Light attack analogous to dezincification.
[d] Severe intergranular corrosion and some attack analogous to dezincification.

only about 1.5 per cent over a length of 1.5 in. All specimens were finished by a process of alternate transverse and longitudinal polishing until the surfaces were sufficiently smooth to permit examination of the structure at a magnification of 100 diameters. During the tests made in air, the specimens were covered with a film of mineral oil.

Because the various materials were intended primarily for use in marine propellers, all corrosion-fatigue tests were made in Severn River water, a brackish estuary water containing one third to one sixth the salinity of sea water, depending on the season and tides. In making these tests, the water was directed onto the test length at the outer fillet; the water was carried back to the inner fillet, thereby enveloping the entire tapered test length. The fatigue values obtained in such a manner are dependent on the specimen size, the speed of rotation, and the length of the run. Consequently, it must be pointed out that the corrosion-fatigue values serve only as guides for comparing the materials and do not represent inherent properties of the alloys.

All fatigue tests were made at 1450 rpm. and were carried out to at least 50 million cycles. As would be expected, most of the castings did not exhibit the soundness of wrought materials, and a considerable number of specimens were required to establish the courses of the stress-cycle graphs. In a few isolated cases the curves could not be located with any great precision, although the best average value has been selected and recorded. Due to the great number of *S-N* curves obtained, it was not considered desirable to present them as a part of this paper.

RESULTS OF TESTS

Results of Mechanical Tests.—The mechanical properties of the alloys are recorded in Table II. Each value is an average of at least two determinations, a sufficient number of specimens having been tested to obtain a representative figure in each instance.

Cavitation-Erosion Tests:

Vibratory hydraulic cavitation tests were made of the several materials to determine the relative resistances to cavitation damage. The actual test work was performed under contract by the Massachusetts Institute of Technology. The apparatus employed the magneto-striction of a nickel tube, vibrated at its natural frequency by a tuned electromagnetic field, to provide the oscillations necessary to produce cavitation (**6, 7**). The tube was made of hard-drawn nickel about 12 in. long, $\frac{5}{8}$ in. in outside diameter, with a 0.028-in. wall thickness. It contained a nickel bushing to hold the specimen at the lower end and a nickel wire for measuring the amplitude of vibration by optical means. The tube was supported in a vertical position by a metal ring silver-soldered around the center of gravity of the whole mass.

The test specimen was $\frac{1}{2}$ in. in diameter and weighed approximately 13.5 g. Prior to testing, the surface to be tested was finished with 00 Manning paper. Two specimens of each material were tested for 20-min. intervals for a total of 120 min. However, materials OV and PJ showed such high resistance to the erosion that they were tested for an additional 60 min. If the results for a pair of specimens did not show a fair agreement, a third specimen was run long enough to show the average behavior of the material.

The cavitation tests were carried out in sea water obtained from Boston Harbor. The following conditions were maintained:

Frequency of vibration, cycles per second	6500 ± 200
Total motion of specimen, mm.	0.074 ± 0.004

Static pressure	Atmospheric
Temperature of sea water, deg. Cent.	25 ± 1 (77 F.)
Air content of water, per cent by volume at S.T.P. (standard conditions of temperature and pressure)	1.3 ± 0.15

The relative cavitation resistance *versus* vibration time for the various samples is presented graphically in Figs. 2, 3, and 4. The relative resistance is the ratio of the weight loss for a standard brass sample to the weight loss for the test material. The reference brass contained approximately 35 per cent zinc and 2 per cent lead. In the case of material OV in Fig. 2, the points were so irregular that the curve is shown as a broken line.

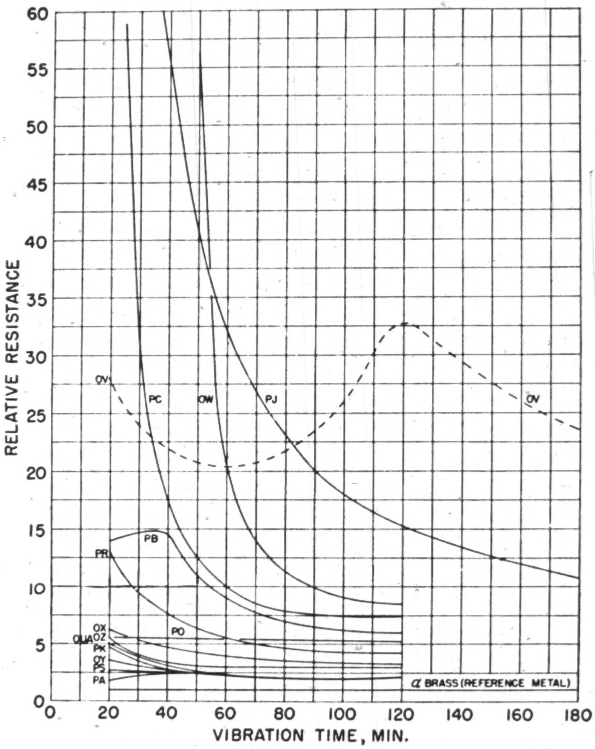

FIG. 2.—Hydraulic Cavitation Tests. Relative resistance *versus* vibration time.

Materials designated by combination of letters. Reference material is alpha brass. Frequency, 6500 cycles per sec. Water from Boston Harbor. Curves are drawn through points obtained at 20-min. intervals.

Corrosion Tests:

Corrosion tests were made in quiescent but constantly changing brackish Severn River water maintained at 80 ± 5 F. The specimens in most cases consisted of $\frac{3}{4}$-in. rounds, 3 in. long, partially suspended in the water. These were kept in corrosion for a period of 515 days. The severest corrosion for the several alloys occurred at or above the water line. Corrosion below the water line was, in general, of the same type, but progressed at a much slower rate. In the extreme right column of Table II are presented the principal types of corrosion occurring for the various compositions.

TREATMENT AND DISCUSSION OF DATA

The variety of alloy compositions included in this investigation was large, thereby making comparison of the many properties a difficult task. The matter is complicated further by the fact that one or two properties alone cannot be set aside as the prime considerations in selecting propeller materials. For example, one engineer may look for alloys with high cavitation resistance. Another may place the greatest weight on good fatigue properties. Still another may insist on relatively high strength combined with good ductility. If the propeller must have thin blades, a high elastic modulus is considered desirable.

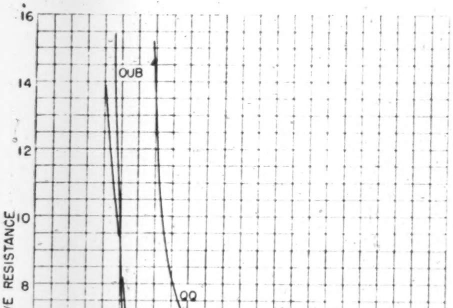

FIG. 3.—Hydraulic Cavitation Tests. Relative resistance *versus* vibration time.

Materials designated by combination of letters. Reference material is alpha brass. Frequency, 6500 cycles per sec. Water from Boston Harbor. Curves are drawn through points obtained at 20-min. intervals.

In view of these facts, it was believed worth while to select materials with good all-around properties, so that these same materials would be adaptable for as many service conditions as possible. In making the selections, the authors have chosen to use an arbitrary system to aid in the discussion and comparison of the several alloys. The system of comparison is shown in Table III, in which the series of ratios are based on the various values for mechanical properties and the corresponding values for cast manganese bronze, material OY. The strength values for this bronze were selected as standards for the reason that the maximum experience for ship propellers has been with this material. The column to the extreme right is an average index number, obtained by averaging the several ratios, and is a rough merit index for each composition.

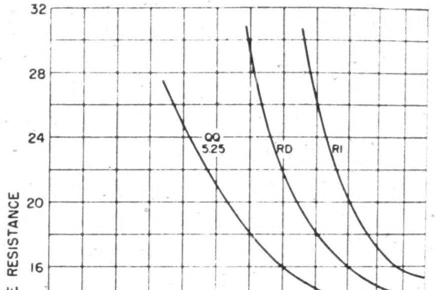

FIG. 4.—Hydraulic Cavitation Tests. Relative resistance *versus* vibration time.

Materials designated by combination of letters. Reference material is alpha brass. Frequency, 6500 cycles per sec. Water from Boston Harbor. Curves are drawn through points obtained at 20-min. intervals.

It may be noted that seven of the alloys possess merit numbers of 2.00 or higher. Among these are the three chromium-nickel austenitic alloys. Material OV had good tensile and elastic properties; however, its high index number was due mainly to the unusually high ratio for cavitation resistance, namely, 12.4 against 1.0 for cast manganese bronze. Even so, the cavitation characteristics of this material were quite erratic. Materials OW and PC, the former cast and the latter forged, have shown uniformly good properties throughout, with the tensile and elastic properties producing the lowest ratios. The high elastic moduli for the austenitic materials are considered desirable for thin blade sections. A disadvantage of the austenitic compositions is their susceptibility to contact corrosion in sea water. These alloys would be most suitable for small propellers, with preference given to material OV, the 16 per cent chromium, 6 per cent nickel composition.

Material RE, a nickel-copper-silicon alloy, has shown excellent tensile strength and elastic properties in both tension and compression. The alloy is relatively hard and has reasonably good ductility and notch impact resistance. The fatigue strength is fairly high. The stiffness of this alloy is comparable to that of the austenitic steels. Material RE has moderately good resistance to hydraulic cavitation.

TABLE III. RATIOS OF MECHANICAL PROPERTIES BASED ON VALUES FOR MANGANESE BRONZE, MATERIAL OY.

Material Designation	Tensile Strength	Proof Stress	Yield Strength	Proportional Limit in Tension	Proportional Limit in Compression	Elongation	Reduction of Area	Charpy Impact	Modulus of Elasticity	Endurance Limit	Corrosion Fatigue	Cavitation Resistance	Average Index Number
QV	1.38	1.66	1.50	1.96	1.29	0.54	0.85	3.43	2.06	2.89	2.33	12.4	2.69
PC	1.08	1.46	1.38	1.50	1.23	2.62	2.88	3.39	2.05	3.39	3.11	3.26	2.28
OW	1.03	1.33	1.56	1.01	1.20	1.88	2.84	3.56	2.06	2.50	1.94	3.70	2.06
RG	1.41	4.10	2.94	5.36	...	0.92	1.88	...	2.06	...	...	1.52	...
QOA	0.38	0.60	0.50	0.77	0.68	0.54	0.46	1.22	1.24	0.74	0.61	0.52	0.69
QOC	0.30	0.54	0.52	0.60	0.61	0.42	0.38	0.56	0.87	0.83	0.67	0.56	0.57
OX	1.08	0.95	1.04	1.10	1.10	0.96	0.85	0.96	1.04	1.00	1.00	1.48	1.05
OY	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00	1.00
OZ	1.17	0.96	1.06	0.90	0.85	1.23	1.08	0.96	1.00	1.13	1.11	1.39	1.07
PA	0.97	0.85	1.10	0.75	0.63	1.42	1.27	0.91	1.02	1.50	1.39	0.74	1.05
PB	1.40	2.10	2.37	2.88	1.66	0.61	0.61	0.56	1.03	1.94	1.83	2.61	1.63
RI	1.51	3.81	3.23	5.08	2.69	0.38	0.46	0.56	1.07	1.78	1.78	6.65	2.42
QU	1.19	1.49	1.50	2.20	1.39	0.61	0.61	0.70	1.04	1.50	1.05	1.52	1.23
OUA	0.68	2.20	1.66	2.68	1.75	0.15	0.23	0.70	1.07	1.67	...	1.00	1.25
OUB	1.00	2.41	1.82	3.08	2.11	0.42	0.69	0.70	1.09	1.02	0.89	1.13	1.36
ADU	0.92	2.19	1.56	4.28	...	0.42	0.65	...	1.05	1.22	0.89	...	1.46
RD	1.20	1.68	1.45	2.44	1.52	0.50	0.46	0.35	1.35	2.55	2.22	6.13	1.82
PJ	1.17	0.87	1.16	0.84	0.48	0.92	0.88	0.96	1.16	2.78	2.61	6.87	1.72
OT	0.68	0.80	0.90	0.55	0.65	0.61	0.58	0.43	1.03	1.67	1.44	1.87	0.93
PK	1.01	1.16	0.98	1.10	1.03	1.85	1.92	1.69	1.68	2.00	1.39	0.74	1.38
PS	0.72	0.94	0.90	0.97	0.94	1.11	1.19	1.13	1.43	1.33	1.11	0.96	1.06
PR	1.26	2.86	2.59	3.79	2.44	0.31	0.73	0.61	1.73	1.67	1.22	1.87	1.76
RE	1.87	5.00	3.68	6.60	4.59	0.38	0.58	1.09	2.01	1.77	1.44	2.61	2.64
PO	0.86	1.91	1.42	1.23	1.36	0.15	0.27	0.48	1.82	2.00	...	2.35	1.26
QQ-525	2.05	4.89		5.66		0.08	0.00	0.13	1.31	1.94	1.50	6.00	2.36
QQ-575	1.63	6.57	...	9.17	6.83	0.04	0.00	0.17	1.40	1.72	1.72	1.83[a]	2.92
QJC	0.44	1.55	...	...	3.41	0.04	0.04	0.56	1.32	0.89	0.86	...	1.01
QY	1.56	6.25	...	...	...	0.04	0.08	...	...	...	...	...	...
QJA	1.47	4.42	...	4.40	4.07	0.08	0.08	0.26	1.15	1.44	1.39	2.48	1.93
QJD	0.67	1.90	...	2.64	2.06	0.08	0.15	0.70	1.33	0.78	0.78	0.78	1.08
AAD	0.75	0.64	0.68	0.77	...	2.00	2.15	...	1.00	0.89	0.28	...	1.02
AAE	0.85	0.37	0.64	0.33	...	1.04	2.12	...	0.55	0.89	...	...	0.85

[a] As received, not heat-treated.

Material RI, a high-strength bronze, has a high tensile strength with good elastic properties in tension and compression. The ductility as determined by the tension test is reasonably good. The modulus of elasticity is slightly higher than manganese bronze. In addition, the fatigue characteristics are considered satisfactory.

The heat-treated copper-beryllium

alloy QQ has shown good strength values. When tempered at 525 F., the material obtained its maximum tensile strength; the higher temper, at 575 F., lowered the tensile strength considerably, but increased the elastic properties. Alloy QQ yielded excellent results in the cavitation test. Among the principal disadvantages of the cast copper-beryllium are the very low ductility and impact values. Furthermore, this alloy would present a difficult welding problem under conditions where it was desired to restore eroded areas by deposition.

There were a few other alloys which might be quite useful, although their merit indexes were not as high as the seven materials discussed above. Material RD, a modified aluminum bronze, showed excellent resistance to cavitation. It had only about one half the elastic property values exhibited by material RI, although it did have a higher modulus of elasticity. Material PS, an aluminum bronze, was good from the standpoint of tensile, ductility, fatigue, and cavitation properties. When exposed to sea water, all three materials, RD, RI, and PS, are subject to dealuminization in a manner analogous to the dezincification of brass. However, they do resist salt-water pitting attack reasonably well.

Other than material QQ, the best of the beryllium alloys was the copper-nickel-beryllium composition QJA. All of the beryllium alloys had low ductilities and reductions of area. It is believed that the alloys containing beryllium were handicapped somewhat by poor casting technique. It is understood that the melting and casting practice has been improved since these test plates were fabricated. Moreover, considerable progress has been made in the preparation of master alloys containing beryllium.

An examination of Table II discloses that the manganese-bronze material CHC, obtained from a $9\frac{1}{2}$-in. thick section of an actual propeller, had just slightly lower properties than the other manganese bronzes cast in plates 2 to $2\frac{1}{2}$ in. thick. This seems to be a good indication that the plates prepared for the investigation yielded properties which would be representative of those encountered in fairly large propellers.

CONCLUSIONS

As is usually the case with wrought materials, no correlation was found to exist between the tensile and salt-water corrosion-fatigue properties of the cast alloys.

Judging from the test results, there does not appear to be any definite correlation between cavitation resistance and hardness. The maximum cavitation resistance was obtained with materials OV, PJ, QQ-525, RD, and RI. Exclusive of the copper-beryllium alloy QQ-525, the Brinell hardnesses of the aforementioned materials ranged from 160 to 232. Material QQ-525 had a Brinell hardness of 368 to 383.

For most of the materials, the proportional limits in tension compared favorably with the corresponding values in compression. Exceptions are noted, particularly in the case of alloys having low elastic properties. In these cases, the values in compression tend to be higher than those in tension.

In selecting a single material from all the alloys tested, the nickel-copper-silicon alloy RE stands out for several reasons: it has high strength values throughout, with fairly good ductility; the alloy is relatively hard but machinable; the notch impact value is good; the resistance to hydraulic cavitation is quite satisfactory, the relative resistance being 2.61 to 1.00 as compared with manganese bronze; the material has shown maximum stiffness as revealed by the elastic modulus; and finally, there exists a background for casting, heat treating, and welding this composition.

The results presented in this paper have been used in an attempt to find superior marine propeller materials, particularly for severe service conditions. However, the fact that some of the alloys had low merit index numbers is not to be construed to mean that those alloys are of little value. Actually, some of them are quite worthwhile materials. For example, the manganese bronzes, with index numbers around 1.0, will remain among the cheapest and most practical compositions for use in the shipbuilding industry.

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PNEUMATIC FATIGUE MACHINES*

BY F. B. QUINLAN[1]

HISTORY

During the war the General Electric Co. was forced to make fatigue tests on gas-turbine buckets, because a correlation between standard test specimens and actual bucket performance was very difficult to obtain. While our experience in this work has been largely with gas-turbine buckets, it would seem that this difficulty applies to a wide variety of parts that are subjected to reversed bending stresses. The buckets have, roughly speaking, a crescent-like cross-section. In precision cast buckets, the metal usually has a finer grain structure at the points of the crescent than in the heavier mid-section. Grain structure has a pronounced effect on the fatigue strength and grain structure is determined by the shape of the cast part; consequently, fatigue tests on actual buckets became a necessity.

Under operating conditions, the buckets are subjected to periodic disturbing forces as they pass through regions of alternately high and low gas pressures. In view of this, it was logical to vibrate these buckets with an interrupted air stream.[2]

FIRST MACHINE

The first design of a pneumatic fatigue testing machine was crude but encouraging. It consisted of welding a bucket *A* to a 1-in. bolt head *B* and bolting it firmly to a heavy steel plate *C* (see Fig. 1). A short rigid piston *D* is mounted on the upper or free end of the bucket. The axis of the piston is at right angles to the centerline of the bucket and lies in the plane of vibration of this centerline. A cylinder *E*, mounted on a vertical support *F*, is coaxial with the piston *D* and is adjustable along a line corresponding to the axis of the piston. There is just sufficient clearance between the piston *D* and the cylinder *E* to prevent contact under operating conditions.

The cylinder is adjusted so that its open end and the face of the piston lie in approximately the same plane. A steady stream of air is admitted to the cylinder and the pressure is increased until the piston is forced away from the cylinder. When this occurs, the gap between the piston and the cylinder increases, which in turn causes the air pressure in the cylinder to drop. The spring action of the bucket, acting as a cantilever, then forces the piston back into the cylinder and the cycle is repeated. It is obvious that the frequency of this cycle is determined by the natural frequency of the cantilever. The amplitude of vibration is governed by the air pressure.

The bucket is heated by placing a cylindrical, resistance-wound furnace around it and closing the upper end with an asbestos cover. The piston support protrudes through an opening in the cover and an air deflector is put under the piston to prevent air from blowing down into the furnace. The furnace temperature is controlled by a thermocouple cemented or spot-welded to the bucket near the region of maximum stress.

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[1] Metallurgical Section, Works Laboratory, General Electric Co., Schenectady, N. Y.

[2] C. F. Jenkins and G. D. Lehman, "High Frequency Fatigue," *Proceedings*, Royal Soc., p. 83 (1929).

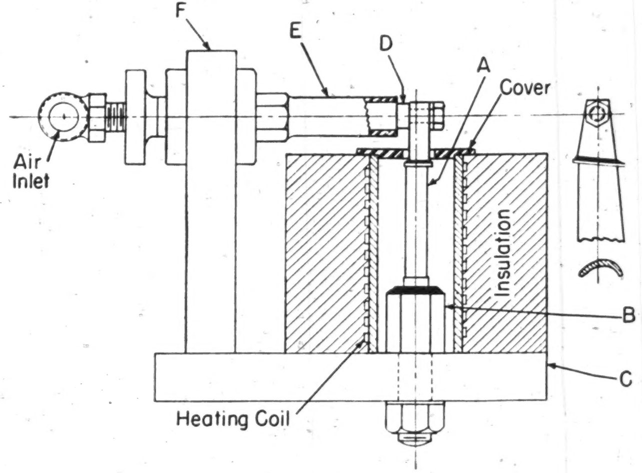

FIG. 1.—Vertical Semisection of Single Cylinder Pneumatic Fatigue Machine.

A—Turbine bucket
B—1-in. bolt
C—Base plate
D—Piston
E—Cylinder
F—Cylinder support

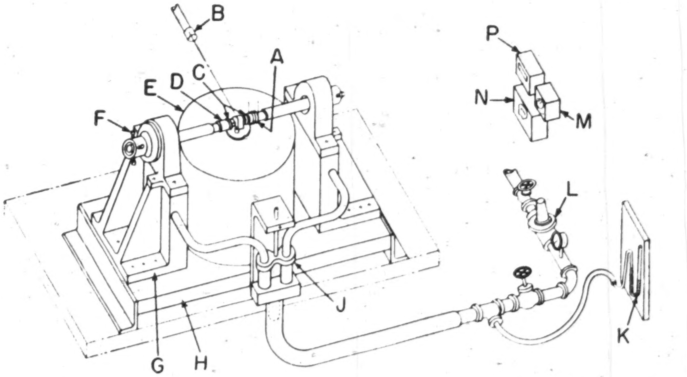

FIG. 2.—Schematic Diagram of Opposed-Cylinder Pneumatic Fatigue Machine.

A—Frequency pick-up coil
B—Amplitude measuring telescope
C—Pistons
D—Adjustable cylinder
E—Furnace
F—Cylinder alignment
G—Pedestal
H—Base plate
J—Half wavelength tuning device
K—Mercury manometer
L—Pressure control valve
M—Frequency indicator
N—Frequency meter
P—Temperature control

The amplitude of vibration is measured optically, and the frequency is indicated by an electronic frequency meter with a magnetic pick-up device.

This machine worked fairly well. The bucket and piston assembly had a natural frequency of approximately 250 cycles per second or 900,000 stress reversals per hour. Stresses were produced up to 80,000 psi., at a total amplitude of 0.200 in. However, when testing at low stresses, below 20,000 psi., the amplitude was difficult to control. The axial position of the cylinder was very critical; also, the amplitude would suddenly increase and then drop back to normal and repeat this in a fairly rhythmic manner. This variation was noticeably in step with a very low-frequency beat note. It was later discovered that this was the result of two frequencies, the resonant frequency of the bucket assembly and the resonant frequency of the air column in the cylinder.

LATER DEVELOPMENTS

In order to make a more stable machine, an opposed cylinder-type driving mechanism was developed. Its operation is the same except that the bucket receives an impulse every half cycle instead of every cycle. In Fig. 2, the two cylinders *D* are connected to a common manifold, and this receives air held at constant pressure, from a pressure control valve *L*. The total air path or the distance from one cylinder mouth to the other (*C* around through *J* to *C*) was made equal to one half a wavelength, at the frequency of the bucket assembly, thereby putting the air impulses 180 deg. out of phase.

This machine will produce stresses up to 100,000 psi. with approximately one fifth the amount of air used in the original machine. The sudden increase in amplitude noticed during the aforementioned beat-note effect has been put to good use. When the air pressure impulses are tuned to come at the precise instant that the piston *C* is entering the mouth of the cylinder *D*, the driving force is increased many times. For instance, at an amplitude producing a stress of 40,000 psi., it is now possible to reduce the air pressure from 20 to 2 psi. The amplitude is remarkably steady and may be varied by adjusting the air pressure. Figure 2 shows the sliding tube arrangement *J* for increasing or decreasing the length of the air path.

In going from ambient temperature to 1350 F., the bucket frequency drops from 250 to approximately 210 cycles per second (this change varies with the alloy), necessitating a change in the total air path as follows:

$$\frac{\text{1100 ft. per sec.**}}{\text{250 cycles per sec.}} = \text{4.4 ft. or 2.2 ft. for } \tfrac{1}{2} \text{ wavelength at 250 cycles per sec.}$$

$$\frac{\text{1100 ft. per sec.}}{\text{210 cycles per sec.}} = \text{5.24 ft. or 2.62 ft. for } \tfrac{1}{2} \text{ wavelength at 210 cycles per sec.}$$

Total increase in air path is 0.42 or 5 in.

An electric furnace has been designed which will allow tests to be made with a bucket temperature of 1700 F.

By altering the design of the support, almost any standard test specimen may be run in the machine.

This type of fatigue testing machine (Fig. 2) has no parts to wear out as nothing moves but the piece to be broken. It is also an extremely easy machine to operate. The cylinders are mounted on ball seats *F* so they may easily be aligned with the pistons. On the end of each cylinder is an adjustable sleeve *D* allowing the operator effectively to back off the cylinder as the amplitude is increased. Mounted on one of these sleeves is a small coil of wire *A* and a magnet used for measuring the frequency.

When the bucket fails, it ceases to vibrate, and this produces a slight increase in the manifold pressure. A mercury manometer *K* responding to this pressure rise closes a contact which in turn operates relays that shut the

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** Measurements of the total air path show that it exceeds the length required for a true half wavelength by a small amount. A thorough investigation of the pressure distribution throughout the tube-length indicates that this added length acts as an impedance matching device. For a maximum transfer of energy, the impedance of the resonant air column should match the impedance of the vibrating cantilever.

machine down and cut off a cycle counting mechanism.

Current work in this field is giving preliminary data which indicate that by using a recording frequency meter, the chart will show what appears to be a point of incipient failure. The chart is also a permanent record of the frequency and time of run.

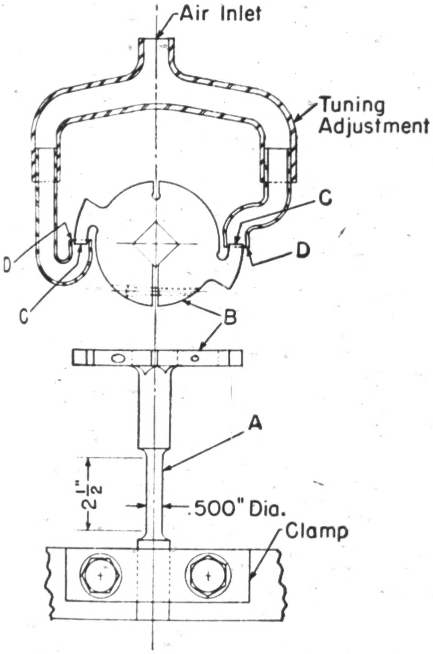

FIG. 3.—Schematic Drawing of Torsional Fatigue Machine.

A—Test specimen
B—Flywheel
C—Piston
D—Cylinder

The curve is flat at a constant frequency for the greater part of the test, but for some time before a visible crack appears the chart reveals a gradual decrease in frequency. The point at which this frequency deviates from a horizontal line is taken as the indication of the point of incipient failure. As may be expected, X-ray and Zyglo tests fail to detect any crack if the piece is examined at this time. However, an increase in the number of stress reversals will cause the crack to grow and later become visible. In some pieces an internal flaw may be the origin of this incipient failure and the crack will take longer to appear at the surface.

The most recent information indicates that brittle materials show this point of incipient failure after 90 to 99 per cent of the total time to fracture, while in some of the more ductile forged samples it may appear at 70 per cent of the total time, depending upon the applied stress. The slope of this decreasing frequency curve may be taken as an indication of the rate of growth of a crack through a particular alloy at a given stress. Samples made from various alloys show an appreciable difference in rate of crack growth. This phenomenon deserves and will receive considerable attention in the future.

TORSIONAL FATIGUE MACHINE

A pneumatic torsional fatigue testing machine for both room and elevated temperatures is shown schematically in Fig. 3. This machine has a very stable driving mechanism, and, again, there are no parts to wear out. The test specimen *A* is a vertical shaft with a small flywheel *B* at the upper or free end. On this flywheel are two pistons *C* diametrically opposite and with their axes on an arc having the same center as the wheel. The cylinders *D* are also formed on this same arc, so that the pistons may enter them without touching the walls. In this case, the operation is the same as the cantilever machine except that the air impulses are in phase rather than 180 deg. out of phase. When two pistons are used, it is important to keep them in phase, otherwise a bending moment is superimposed on the torsion. It is, however, possible to use two sets of opposed pistons diametrically opposite each other and then have the cylinders adjusted to one half a wavelength.

The test specimen in Fig. 3 has a 0.500-in. diameter by 2.5-in. long test section. The natural frequency of the test assembly is 230 cycles per second. The angular displacement is read optically and the frequency electrically.

FATIGUE OF THIN STRIP

A miniature fatigue machine has been built. It is of the opposed cylinder type, but, as it is designed for vibrating strip 0.002 in. thick, it cannot have pistons because they would reduce the natural frequency to too low a value. Therefore, this is designed so as to have the strip act as its own piston. This machine has operating frequencies of from 300 to 1200 cycles per second, thus making the time of testing very short. Ten million stress reversals may be obtained in less than an 8-hr. day.

The test specimen for this unit is a 0.25-in. wide strip of the required thickness; the length may vary from 0.5 to 1.25 in., depending upon the frequency desired. It is not necessary, therefore, to use a specially machined sample.

DISCUSSION

Mr. G. A. Olsen.[1]—Where you said it was possible to reverse completely the stress cycle on that machine, it seemed to me as though the pistons on opposite sides of the specimen would direct the air pressure so that it would twist only in one direction.

Mr. F. B. Quinlan (*author*).—In the torsional fatigue machine illustrated, it is true that there is a slight steady torsional stress superimposed on the vibratory stress, but this is completely eliminated in the four-cylinder machine.

Mr. D. B. Shotwell.[2]—At Caterpillar a system of automatic amplitude control is in use. The system operates slightly below resonance and changes in amplitude are brought about by changes in frequency of the exciting system.

Mr. Quinlan.—In the majority of cases automatic amplitude control is not necessary because, unlike resonance in an electrical circuit, the resonant peak in this pneumatic circuit is quite broad and flat; however, it is quite possible to install an automatic amplitude control device without going to much expense or trouble.

Mr. W. T. Bean.[3]—How did you measure the frequency at operating temperature?

Mr. Quinlan.—Frequency is measured by placing a magnetic pickup device close to the steel pistons. This gives us an a-c. output at the frequency of the vibrating part. This signal is indicated on a General Radio frequency meter.

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[1] Bakelite Corp., Bloomfield, N. J.

[2] Research Engineer, Caterpillar Tractor Co., Peoria, Ill.

[3] Research Engineer, in charge of Experimental Stress Analysis, Continental Aviation and Engineering Corp., Detroit, Mich.

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON TESTING OF PARTS AND ASSEMBLIES

The Symposium on Testing of Parts and Assemblies was held in Buffalo, N. Y., June 26, 1946, during the Forty-Ninth Annual Meeting of the Society. The Symposium was sponsored jointly by the American Society for Testing Materials, through its Administrative Committee on Simulated Service Testing, and the Society for Experimental Stress Analysis. The Symposium Committee was as follows: R. L. Templin, Chairman, Aluminum Company of America; Charles Lipson, Chrysler Corp. (President, S.E.S.A.); J. M. Frankland, Chance-Vought Aircraft Division of the United Aircraft Corp.; E. W. Upham, Chrysler Corp.; and P. G. McVetty, Westinghouse Electric Corp.

The Administrative Committee on Simulated Service Testing is charged with the obligation of resolving many of the differences between present test methods and service performance. It is the aim of the Society, through the committee, to promote through group discussions, and symposia, a more thorough knowledge and realization of our exact status in the correlation of testing and serviceability, to the end that whatever test methods finally result—be they laboratory tests, simulated service, or actual service tests—we will be assured of their validity.

This symposium is one of a series which have been planned, and since in this case the Society for Experimental Stress Analysis was directly concerned with the subjects being discussed, they were invited to join with A.S.T.M. as co-sponsors.

The Symposium consists of the following papers:

Introduction—L. L. Wyman
The Fatigue Strength of Lap Joints in Some Magnesium Sheet Alloys—H. J. Grover and L. R. Jackson
Fatigue Characteristics of Magnesium Castings—George H. Found
Endurance—A Criterion of Design—W. T. Bean
Pneumatic Fatigue Machines—F. B. Quinlan
Automotive Rear Axles and Means of Improving Their Fatigue Resistance—O. J. Horger and C. H. Lipson
Stress Concentration and the Fatigue Strength of Engine Components—Charles Gadd, N. A. Ochiltree, and Andrew Zmuda

The Symposium was held in two sessions with L. L. Wyman, General Electric Co. (Chairman, Administrative Committee on Simulated Service Testing), as technical chairman of the morning session and Charles Lipson (President, Society for Experimental Stress Analysis) presiding at the afternoon meeting.

These papers together with discussion have been issued by the A.S.T.M. as Technical Publication No. 72 entitled, "Symposium on Testing of Parts and Assemblies."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON TESTING OF BEARINGS

The papers and discussions included in this session were presented as a Symposium on Testing of Bearings, held at the Forty-Ninth Annual Meeting of the Society in Buffalo, N. Y., June 24, 1946.

The Symposium was sponsored by the Administrative Committee on Simulated Service Testing, through a special committee consisting of E. W. Upham, Chrysler Corp., Chairman; J. M. Frankland, Chance-Vought Aircraft Division of United Aircraft Corp.; and K. D. Williams, Bureau of Ships, U. S. Navy.

The current interest in parts testing makes this a timely symposium. Since current trends in design call for economical uses of materials, with the corresponding elimination of high factors of safety, testing engineers must devise means of checking the manufactured product. Life testing of plain and antifriction bearings is representative of the practice of judging and comparing materials by testing parts and assemblies. Over a period of years, comparison of the results of such tests with actual field service has built up such a wealth of correlative background that we can rely quite heavily on these specialized forms of laboratory testing.

The six papers—which cover descriptions of the specialized test machines used, procedures that have been developed, and the results obtained—were presented by leading engineers in the field as follows:

Introduction—E. W. Upham
Life Testing of Plain Bearings for Automotive Engines—E. T. Johnson
Fatigue Testing Machines for Ball and Roller Bearings—Thomas Barish
Metallographic Observations of Ball Bearing Fatigue Phenomena—A. B. Jones
Fatigue Testing of Roller Bearings—H. R. Gibbons
Testing of Bearings Under Controlled Load—J. M. Frankland and C. B. Innes

The chairman of the session was J. R. Townsend, President of the Society, and Materials Engineer, Bell Telephone Laboratories, Inc., New York, N. Y.

The papers together with discussion have been issued by the Society as a separate publication entitled "Symposium on Testing of Bearings."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON MATERIALS FOR GAS TURBINES

The papers and discussions included in the Symposium on Materials for Gas Turbines were presented at the Thirteenth and Seventeenth Sessions of the Forty-Ninth Annual Meeting of the Society, held in Buffalo, N. Y., June 24–28, 1946. These two sessions were sponsored by the Joint Committee on the Effect of Temperature on the Properties of Metals. While the last three papers of the symposium do not cover gas turbine materials specifically, the committee in charge felt that because of their related interest they should be included in the Symposium, as well as in the *Proceedings.*

The coordination and interchange of information and ideas relating to the behavior of metals at high and low temperatures has long been one of the important functions of the Joint Research Committee. The committee has endeavored to be of service by promoting discussions and presenting data as early as possible in new fields of interest and application. During the war period, the development of the turbosupercharger, jet engine, and gas turbine for aircraft, rockets of all kinds, and gas turbines for ships or other purposes, involved the creation and development of many new high-temperature alloys. Practically all of this development was held as secret, confidential, or restricted, and was carried out by the Government Services, or under contract with these services.

Many of the members of the Joint Research Committee, and others long associated with its work, were engaged wholly or in part in these developments. Most of the high-temperature testing facilities of the country were assigned to this research work.

The committee believed that it would render a useful service to all interested in such fields of application by arranging for a technical session devoted to a consideration of these new high-temperature alloys.

The Symposium consists of the following nine papers:

Introduction—N. L. Mochel
Heat-Resisting Metals for Gas Turbine Parts—Howard C. Cross and Ward T. Simmons
High-Temperature Alloys Developed for Aircraft Turbosuperchargers and Gas Turbines—J. W. Freeman, E. E. Reynolds, and A. E. White
Chromium-Base Alloys—Robert M. Parke and Frederick P. Bens
Metallurgy of High-Temperature Alloys Used on Current Gas Turbine Designs—F. S. Badger, Jr. and W. O. Sweeney, Jr.
Alloys and Ceramic Materials for High-Temperature Service—Howard C. Cross
Resistance of Iron-Nickel-Chromium Alloys to Corrosion in Air at 1600 to 2200 F.—Anton deS. Brasunas, James T. Gow, and Oscar E. Harder
Compressive Properties of Aluminum Alloy Sheet at Elevated Temperatures—Alan E. Flanigan, Leslie F. Tedsen, and John E. Dorn
Tensile and Creep Strengths of Some Magnesium-Base Alloys at Elevated Temperature—A. A. Moore and J. C. McDonald

The chairman at the thirteenth session was N. L. Mochel, chairman of the Joint Committee sponsoring the symposium, and Manager, Metallurgical Engineering, Westinghouse Electric Corp., Philadelphia, Pa. At the seventeenth session the presiding officer was J. W. Bolton, former secretary of the Joint Committee, and Director of Metallurgical Research and Testing, The Lunkenheimer Co., Cincinnati, Ohio.

These papers together with discussion are available as a separate publication entitled, "Symposium on Materials for Gas Turbines."

THE EFFECT OF CARBIDE SPHEROIDIZATION UPON THE RUPTURE STRENGTH AND ELONGATION OF CARBON-MOLYBDENUM STEEL*

By S. H. Weaver[1]

Synopsis

Changes in properties of steel for high-temperature equipment maintained at stresses and temperatures affect the design stresses and service life. Specimens from a plate of carbon-molybdenum steel were treated to obtain twelve different conditions of the steel by varying the heat treatment and the amount of spheroidization. Each condition of the steel has been previously[2] tested in long-time creep at 900 and 1000 F. In this investigation, using the same plate of steel, specimens in the twelve different conditions were subjected to long-time rupture-stress and elongation tests at 900 and 1000 F.

As a result of the tests the following conclusions were reached:

1. There is a tendency for all the conditions of the steel to approach a common, stable rupture stress.
2. During a long service life the tested rupture stress for a period of 10,000 hr. does not decrease nearly so much as the creep stress.
3. The rupture elongations for all conditions of the steel except those with initially spheroidized carbides rapidly drop with time.
4. Extrapolation for rupture stress and elongation over the 10,000 to 100,000-hr. test period is questionable.
5. The creep-stress conclusion[2] that 1000 F. is near the practical limit in design stress is not invalidated by data in this paper.

The object of the tests presented in this paper was to obtain data bearing on the service life of high-temperature equipment, particularly steam turbines. The higher operating temperatures produce slow changes in the steel during the service life of the apparatus. One of these changes is due to the spheroidization of the carbides. Tests showing the effect of carbide spheroidization upon the creep strength of carbon-molybdenum steel at 900 and 1000 F. were presented before the Society in 1941.[2] The rupture-stress tests described in this paper were made from the same plate of material that was used for the creep tests.

Plan of Tests

To facilitate comparison of results of the previous creep tests[2] with the present rupture tests, the same plan is used in presenting data. Figure 1, reproduced from the previous paper, is a diagram of the plan of tests. The key letters distinguishing the twelve conditions of the steel and the corresponding creep-test item numbers, both given in Fig. 1, are used for the rupture tests. The order of presentation of the present paper follows, as nearly as possible, the order of presentation of the previous paper on creep tests.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] Turbine-Generator Engineering Div., General Electric Co., Schenectady, N. Y. (Retired)

[2] S. H. Weaver, "The Effect of Carbide Spheroidization Upon the Creep Strength of Carbon-Molybdenum Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 608 (1941).

MATERIAL

The material used in this investigation is a weldable 0.5 per cent molybdenum steel with a chemical composition of 0.17 per cent carbon, 0.42 per cent molybdenum, 0.88 per cent manganese, 0.20 per cent silicon, 0.004 per cent aluminum, and 0.021 per cent Al_2O_3. It was incorrectly stated in the author's previous paper[2] that the steel was "aluminum killed," whereas in reality the heat (No. 57,328) was "silicon deoxidized," and no aluminum was added at any time to the molten steel. While this was a commercial heat of open-hearth, silicon-killed steel, the material was under special observation at all time because of its important application. The steel was made by the Carnegie-Illinois Steel Corp., rolled into plates 4¼ in. in thickness, and supplied to the Babcock & Wilcox Co. for fabrication. The plate was formed into open-end cylinders, 48 in. in inner diameter, by one axial weld. The cylinders, placed end to end, were circumferentially welded to form a fabricated, mercury-vapor, power-boiler drum. The General Electric Co. furnished the mercury-vapor turbine and other apparatus to the Public Service Co. of New Jersey for installation in the Kearny, N. J., power station. After being in operation for 17,939 hr. at 975 F., with fluctuations up to 990 F., 3-in. diameter plugs were trepanned through the boiler walls for examination of welds and base metal. The plates were originally heat-treated before installation by normalizing from 1620 F. and by stress-relieving three times during fabrication, at temperatures ranging from 1030 to 1200 F., for a total of 20 hr. One part of the drum was annealed instead of air cooled. No graphite was found in the plugs trepanned from the normalized or annealed materials, and only a small degree of carbide spheroidization was present. The McQuaid-Ehn test was normal, showing a grain size of No. 3 to 4 with some No. 2 grain size present.

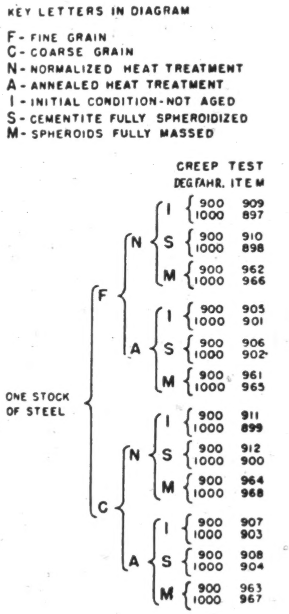
KEY LETTERS IN DIAGRAM
F - FINE GRAIN
C - COARSE GRAIN
N - NORMALIZED HEAT TREATMENT
A - ANNEALED HEAT TREATMENT
I - INITIAL CONDITION - NOT AGED
S - CEMENTITE FULLY SPHEROIDIZED
M - SPHEROIDS FULLY MASSED
CREEP TEST
DEG FAHR.
ITEM
ONE STOCK OF STEEL
F
N
I 900 909
1000 897
S 900 910
1000 898
M 900 962
1000 966
A
I 900 905
1000 901
S 900 906
1000 902
M 900 961
1000 965
C
N
I 900 911
1000 899
S 900 912
1000 900
M 900 964
1000 968
A
I 900 907
1000 903
S 900 908
1000 904
M 900 963
1000 967

FIG. 1.—Plan for Rupture Tests (Same as Plan Used for Creep Tests Reported in 1941).

The deoxidization of the steel requires emphasis because of the large amount of aluminum oxide found in the material. Some tests with aluminum alloy in wrought steel indicate that 1 per cent aluminum in solution is the strongest of the alloy accelerators forming nodular graphite. Austin and Fetzer[3] conclude that while the effect of aluminum oxide is markedly to lower the stability of the iron carbide, the mere presence of the oxide does not suffice; form or degree of dispersion is the important factor.

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[3] Charles R. Austin and M. C. Fetzer, "Cementite Stability and Its Relation to Grain Size, Abnormality and Hardenability," Am. Soc. Metals, Preprint No. 29 (1940).

The separation for chemical analysis of the oxide and the aluminum in solution was at one time uncertain; however, the interested laboratories have now agreed upon and use a satisfactory method of chemical analysis. Three of the aforementioned companies, using metal from different locations in this heat of steel, made six analyses which average 0.0036 per cent of aluminum alloy and 0.0236 per cent of aluminum oxide. The range of the oxide on check analyses was 0.008 to 0.041 per cent, which indicates an uneven or spotty distribution of the oxide in the steel.

TABLE I.—HEAT TREATMENT, PHYSICAL PROPERTIES AND RESULT OF CREEP TESTS AT 900 F.

Key[a]	Item	Heat Treatment[b] (All Specimens Drawn 1200 F.—4 hr.—F.C.)	Spheroidized at 1292 F., hr.	Physical Properties Before and After Creep								Creep Stress for Various Rates per 100,000 hr., psi.		
				Time	Tensile Strength, psi.	Elastic Limit, psi.	Elongation in 2 in., per cent	Reduction of Area, per cent	Rockwell Hardness, "B" Scale	Charpy Impact, Keyhole Notch, ft-lb.				
										Room Temperature	900 F.	10%	1%	0.1%
FNI.........	909	1560 F.—8 hr.—A.C.	Draw only	Before	68 100	39 000	35.5	64.4		36.9				
				After	70 600	46 800	36.4	64.1	75.5	27.7	28.5	27 000	20 000	15 000
FNS.........	910		55	Before	63 200	37 000	36.0	67.0		25.6				
				After	63 500	35 200	35.0	60.5	70.5	18.2	12.2	12 700	9 200	6 600
FNM.........	962		1500	Before	57 700	26 000	36.5	59.4	55.0	9.0				
				After	56 100	30 200	37.2	60.0		2.1	19.5	10 000	8 100	6 400
FAI.........	905	1560 F.—8 hr.—F.C.	Draw only	Before	67 100	35 000	34.5	58.0		26.3				
				After	65 400	37 400	37.1	59.0	72.0	19.5	20.1	16 600	14 100	12 500
FAS.........	906		180	Before	63 700	35 000	36.0	63.0		20.8				
				After	60 600	36 500	38.4	62.9	69.0	5.1	20.1	12 700	10 200	8 000
FAM.........	961		1500	Before	60 200	26 000	33.5	62.2	60.1	8.5				
				After	59 600	29 500	37.2	71.4		5.1	11.1	10 600	8 800	7 000
CNI.........	911	1750 F.—8 hr.—A.C.	Draw only	Before	70 900	41 000	31.5	63.2		24.0				
				After	79 000	31 400	32.9	56.7	84.5	18.8	11.1	30 000	24 300	19 700
CNS.........	912		55	Before	67 200	43 000	33.0	68.4		37.8				
				After	63 400	37 400	39.3	68.9	72.0	20.8	18.2	21 000	16 500	13 000
CNM.........	964		1500	Before	56 900	23 000	36.0	58.9	59.5	2.6				
				After	58 100	31 200	40.7	58.3			19.5	8 800	7 200	5 900
CAI.........	907	1750 F.—8 hr.—F.C.	Draw only	Before	63 600	32 000	34.0	56.9		22.8				
				After	65 500	29 100	37.8	55.6	72.0	16.3	18.2	16 600	13 800	11 600
CAS.........	908		180	Before	61 900	26 000	35.0	60.8		2.5				
				After	62 400	28 100	34.9	60.4	68.0	3.8 2.1	9.0	14 000	10 000	7 250
CAM.........	963		1500	Before	60 400	26 000	35.0	59.4	64.0	2.6				
				After	57 100	30 200	36.4	61.7		2.6	19.5	12 000	8 400	6 400

[a] *F* = Fine Grain Size. *C* = Coarse Grain Size. *N* = Normalized. *A* = Annealed. *I* = Initial, Not Aged. *S* = Spheroidized. *M* = Massed Cementite.

[b] Heat Treatment: F.C. = Furnace Cooled. A.C. = Air Cooled.

Aluminum in solution does not affect grain growth with heat-treating temperature, while the aluminum oxide is used in the steel-making process to control grain size by restraining the grain growth. The steel used in the mercury-boiler drum and in the present creep and rupture tests has a grain diameter-heat-treating temperature relationship approaching the characteristics of a silicon-killed steel. The large amount of aluminum oxide present must exist in an unusual form since it does not restrain the grain growth. With no aluminum added to the melt the oxide must have been introduced by the scrap steel in the furnace charge. This conclusion agrees with foundry experience at the General Electric Co. where specifications require a medium size of the pearlitic or structural grain for the 1 per cent molybdenum castings used at higher operating temperatures. Thirty-five-ton basic, electric arc furnaces are used with a boiling cycle. No aluminum additions are made at any time. Chemical analyses show similar large amounts of aluminum oxide with a negligible restraint upon the grain size.

TABLE II. HEAT TREATMENT, PHYSICAL PROPERTIES AND RESULT OF CREEP TESTS AT 1000 F.

Key[a]	Item	Heat Treatment[b] (All Specimens Drawn 1200 F.—4 hr.—F.C.)	Spheroidized at 1292 F., hr.	Physical Properties Before and After Creep								Creep Stress for Various Rates per 100,000 hr., psi.		
				Time	Tensile Strength, psi.	Elastic Limit, psi.	Elongation, in 2 in., per cent	Reduction of Area, per cent	Rockwell Hardness "B" Scale	Charpy Impact, Keyhole Notch, ft-lb.		10%	1%	0.1%
										Room Temperature	1000 F.			
FNI	897	1560 F.—8 hr.—A.C.	Draw only	Before	68 100	39 000	35.5	64.4		36.9				
				After	69 500	45 600	30.0	60.8	74.5	29.1	15.1	20 000	7 200	2 500
FNS	898		55	Before	63 200	37 000	36.0	67.0		25.6				
				After	63 500	33 200	35.7	64.6	69.5	22.8	30.8	6 000	3 600	1 850
FNM	966		1500	Before	57 700	26 000	36.5	59.4	55.0	9.0				
				After	56 100	32 300	39.5	60.0	61.0	3.4	18.8	4 500	2 700	1 780
FAI	901	1560 F.—8 hr.—F.C.	Draw only	Before	67 100	35 000	34.5	58.0		26.5				
				After	67 000	36 400	35.0	60.9	60.9	19.5	28.5	17 500	7 600	2 900
FAS	902		180	Before	63 700	35 000	36.0	63.0		20.8				
				After	59 500	35 300	37.8	65.5	66.5	7.0	25.6	6 300	3 400	1 850
FAM	965		1500	Before	60 200	26 000	33.5	62.2	60.0	8.5				
				After	59 200	31 200	40.6	61.5	64.0	5.1	26.5	4 700	3 100	2 050
CNI	899	1750 F.—8 hr.—A.C.	Draw only	Before	70 900	41 000	31.5	63.2		24.0				
				After	67 600	35 300	38.8	61.6	76.5	24.0	30.8	22 000	11 000	3 800
CNS	900		55	Before	67 200	43 000	33.0	68.5		37.8				
				After	58 600	39 500	37.9	67.0	76.0	10.0	31.7	9 600	6 400	2 450
CNM	968		1500	Before	56 900	23 000	36.0	58.9	59.5	2.6				
				After	56 600	31 200	37.8	61.5	60.0	1.8	18.8	4 500	2 500	1 100
CAI	903	1750 F.—8 hr.—F.C.	Draw only	Before	63 600	32 000	34.0	56.9		22.8				
				After	66 600	27 000	32.5	55.6	72.5	14.5	18.2	14 200	10 800	3 500
CAS	904		180	Before	61 900	26 000	35.0	60.8		2.5-3.8				
				After	62 400	27 000	34.0	61.6	67.0	2.6	35.5	6 400	4 350	2 100
CAM	967		1500	Before	60 400	26 000	35.0	59.4	64.0	2.6				
				After	60 600	31 200	37.1	59.8	63.5	2.6	19.5	4 900	2 900	1 650

[a] *F* = Fine Grain Size. *C* = Coarse Grain Size. *N* = Normalized. *A* = Annealed. *I* = Initial, Not Aged. *S* = Spheroidized. *M* = Massed Cementite.

[b] Heat Treatment: F.C. = Furnace Cooled. A.C. = Air Cooled.

CARBIDE SPHEROIDIZATION

The degree of the spheroidization of the carbides must be defined to a definite end standard of measurement so that it can be duplicated in tests by other laboratories. Condition S in Fig. 1 is with the cementite fully spheroidized. It is obtained by heating the annealed steel, under no stress, at a constant temperature below the Ac_1 transformation and examining the microstructure at different periods of time. The number

TABLE III.—HEAT TREATMENT AND RESULT OF RUPTURE TESTS AT 900 F.

Key[a]	Item	Heat Treatment[b] (all specimens drawn 1200 F.—4 hr.—furnace cooled)	Spheroidized at 1292 F, hr.	Stress for Rupture, psi.				Elongation in 4 in., per cent				Stress for Total Extension of 1 per cent, psi.				Rupture Curve Slope Change psi.-hr.	Longest Test, hr.
				100 hr.	1000 hr.	10,000 hr.	100,000 hr.	100 hr.	1000 hr.	10,000 hr.	100,000 hr.	100 hr.	1000 hr.	10,000 hr.	100,000 hr.		
FNI	909	1560 F.—8 hr.—A.C.	None	52 000	50 000	46 000	38 000	27	20	11		42 000	36 000	30 000	26 000	50 000–3500	16 100
FNS	910		55	34 000	30 000	24 000	20 000	34	33	23						25 000–1000	7 400
FNM	962		1500	32 000	28 000	24 000	20 000	35	31	28						No change	5 600
FAI	905	1560 F.—8 hr.—F.C.	None	46 000	41 000	32 000	25 000	28	20	8						44 000- 660	17 400
FAS	906		180	34 000	30 000	25 000	23 000	31	31	25						No change	6 400
FAM	961		1500	32 000	28 000	24 000	21 000	31	31	28		21 000				No change	11 500
CNI	911	1750 F.—8 hr.—A.C.	None	54 000	53 000	52 000	50 000	25	15	7						No change	4 000
CNS	912		55	40 000	37 000	34 000	31 000	21	20	16		34 000	33 000	31 000	29 000	No change	5 600
CNM	964		1500	34 000	29 500	26 000	23 000	31	29	27						No change	6 800
CAI	907	1750 F.—8 hr.—F.C.	None	46 000	40 000	34 000	28 000	32	17	8		26 000	24 000	22 000		45 000–250	12 200
CAS	908		180	32 000	29 000	24 000	20 000	31	28	16						27 500–1800	5 000
CAM	963		1500	30 000	27 000	23 000	20 000	31	30	23						No change	14 000

[a] F = Fine grain size. C = Coarse grain size. N = Normalized. A = Annealed. I = Initial, not aged. S = Spheroidized. M = Massed cementite.

[b] Heat treatment: F.C. = Furnace Cooled. A.C. = Air Cooled.

TABLE IV.—HEAT TREATMENT AND RESULT OF RUPTURE TESTS AT 1000 F.

Key[a]	Item	Heat Treatment[b] (all specimens drawn 1200 F.—4 hr.—furnace cooled)	Spheroidized at 1292 F., hr.	Stress for Rupture, psi.				Elongation in 4 in., per cent				Stress for Total Extension of 1 per cent, psi.				Rupture Curve Slope Change, psi -hr.	Longest Test, hr.
				100 hr.	1000 hr.	10,000 hr.	100,000 hr.	100 hr.	1000 hr.	10,000 hr.	100,000 hr.	100 hr.	1000 hr.	10,000 hr.	100,000 hr.		
FNI	897	1560 F.—8 hr.—A.C.	None	38 000	30 000	16 000	8 800	24	20	15		27 000	24 000	13 500	6 500	34 000- 700	7 400
FNS	898		55	22 000	16 500	11 000	7 000	43	36	41		14 000	10 000	7 700	5 700	18 000- 650	4 600
FNM	966		1500	21 500	16 000	11 000	7 500	48	42	40		12 000	9 000	7 000	5 000	16 000–1300	5 600
FAI	901	1560 F.—8 hr.—F.C.	None	32 000	27 000	16 000	8 800	15	11	15		22 000	20 000	13 000	8 000	28 000- 900	7 300
FAS	902		180	20 000	16 000	11 500	9 000	43	37	45		14 000	10 000	7 500	5 500	No change	4 900
FAM	965		1500	16 000	13 000	10 500	8 600	55	50	39		7 800	...	...	...	No change	16 700
CNI	899	1750 F.—8 hr.—A.C.	None	46 000	37 500	22 000	12 000	18	8	6		38 000	37 500	20 000	11 000	48 000- 110	4 800
CNS	900		55	26 000	20 000	15 000	12 000	32	28	19		20 000	17 000	14 000	11 000	25 000- 220	7 300
CNM	968		1500	21 000	15 000	10 000	7 000	40	50	40		12 000	8 700	6 500	...	18 000- 270	5 800
CAI	903	1750 F.—8 hr.—F.C.	None	36 000	28 000	18 000	12 000	16	9	6		23 000	21 000	19 000	17 000	38 000- 370	6 200
CAS	904		180	20 000	15 000	11 000	8 000	42	36	25		13 000	10 000	8 100	6 500	No change	13 000
CAM	967		1500	18 000	13 000	9 600	7 000	47	49	29		11 000	8 200	6 400	5 000	No change	6 800

[a] F = Fine grain size. C = Coarse grain size. N = Normalized. A = Annealed. I = Initial, not aged. S = Spheroidized. M = Massed cementite.

[b] Heat treatment: F.C. = Furnace Cooled. A.C. = Air Cooled.

of hours of heating required to eliminate the last trace of discernible lamellae formation is considered the end standard for full spheroidization. The normalized steels are said to be fully spheroidized when the spheroids are of the same size as those obtained in the annealed standard for the same heat of steel.

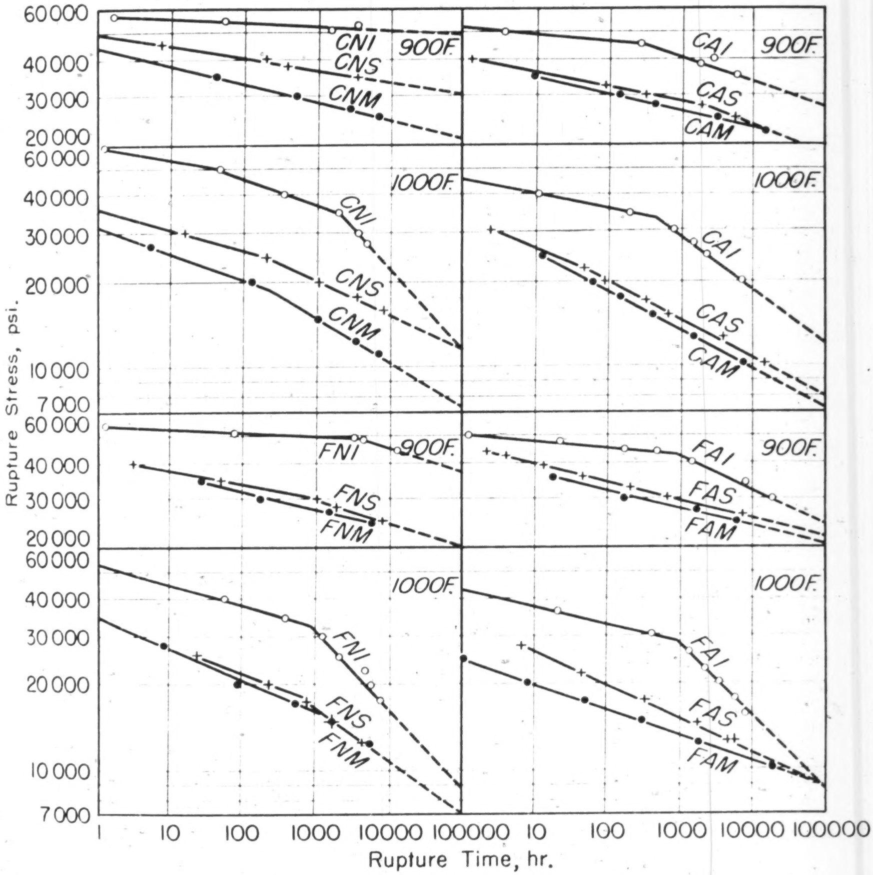

FIG. 2.—Rupture Stress and the Corresponding Time for 0.5 per cent Molybdenum Steel with Differently Spheroidized Microstructures.

For condition M with the massed spheroids, the aim was to continue the spheroidizing time until the carbides were fully coalesced, leaving the ferrite matrix clear. While the expected result was obtained in only one sample, FNM, after 1500 hr. at 1292 F., the later creep and rupture tests show only a small difference between the conditions S and M.

RUPTURE TESTS

A series of rupture-stress tests were made on each of the twelve conditions of the steel in Fig. 1, at 900 F., and upon duplicate bars at 1000 F. The equipment employed is described in previous papers.[4,5] The test specimens were machined to a diameter of 0.253 in. with

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[4] R. H. Thielemann and E. R. Parker, "Fracture of Steels at Elevated Temperatures After Prolonged Loading," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 135, pp. 559–582 (1939).

[5] R. H. Thielemann, "Some Effects of Composition and Heat Treatment on the High Temperature Rupture Properties of Ferrous Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 788 (1940).

enlarged, threaded ends. Elongation measurements are taken over a 4-in. gage length. Elongation-time data were recorded for the constant loads on each of the 140 rupture-stress specimens. It is impractical to reproduce this number of curves; therefore, readings from plots at different periods of time are tabulated in Table III for the tests at 900 F. and in Table IV for the tests at 1000 F. Tables I and II contain data for the corresponding creep tests on the same material given in the previous paper.[2] Key letters from Fig. 1 indicate the condition of the steel in each of the series of tests and the item numbers for the rupture-stress series correspond to those used for the creep tests. The room-temperature physical properties and the before-test microstructures at a magnification of 100 and 1500 are given in the author's previous creep-test report.[2]

Tables III and IV give interpolated and, whenever possible, extrapolated data on the rupture stress, elongation at rupture, stress for an extension of 1 per cent, the stress and time coordinate where the curve of *log rupture stress versus log time* changes slope, and the longest test in each rupture series.

Figure 2 shows the relation between constant-load rupture stress and rupture time for each of the specimens tested in the twelve conditions at 900 and 1000 F. There is a spread between the rupture-stress curves for the initial and spheroidized conditions similar to that found in Fig. 14 of the creep tests previously reported.[2] A change in slope occurs in all curves for the initial condition *I* of the steels, but the amount of the change is greatly reduced for the normalized spheroidized conditions and is not present within the 10,000 hr. of test for the annealed spheroidized steels.

The change in the slope of curves through the rupture-stress test points is ascribed[4] to oxidation of specimens by the air in the test furnace. Oxygen penetrating the metal increases the slope of the curve. A change in the type of fracture is evident where the slope of the curve increases. Failures in the shorter time tests are characterized by a transcrystalline fracture through the grains, changing in the longer time tests to an intergranular break following the grain boundaries. The later fracture is characterized by a small reduction in area and is usually termed a brittle failure. Tests by Thielemann and Parker,[4] made in a pure, dry hydrogen atmosphere show a similar change in the type of fracture for longer time tests but without the change in slope in the stress-time curve. In the hydrogen gas, intergranular oxidization is absent, so it seems likely that a precipitation of carbides or impurities, or an atomic lattice disturbance at the grain boundaries, influences the change in type of fracture, which is then accelerated by oxygen penetration. This generalized explanation has been confirmed by previous tests[4,5] and the same laboratory equipment was used for the present series of rupture tests.

In Fig. 2 the curves for the spheroidized normalized steels, NS and NM, have a very slight change in slope while the curves for spheroidized annealed steels, AS and AM, have no slope change in tests up to 10,000 hr. This indicates that the effect of oxidization from the furnace air is very slight for the S and M spheroidized conditions and that the change from transcrystalline to intergranular fracture is delayed by the previous spheroidization treatment given the test specimens.

In Fig. 3 the percentage elongation in 4 in. at the rupture period is plotted for each test specimen shown in Fig. 2. All tests in the initial condition *I* show a rapid decrease in the elongation, which accompanies the change to an intergranular fracture. In both of the spheroidized conditions, S and M, the elongations are, and remain, at high values. The intergranular fracture appears in some of the later and longest time test specimens.

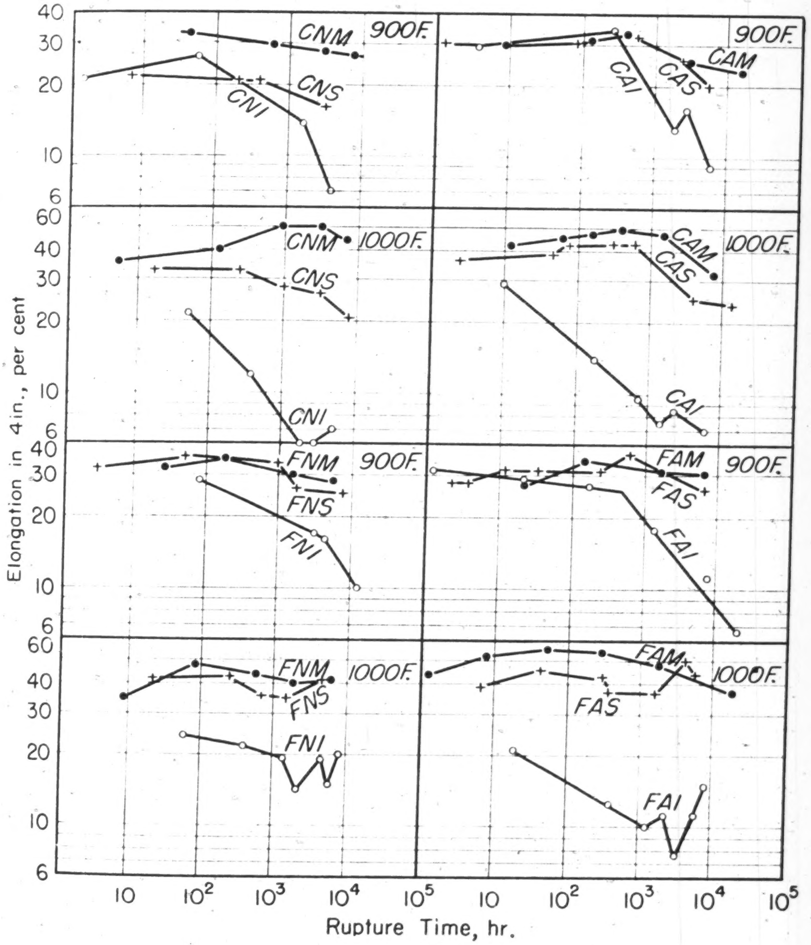

FIG. 3.—Elongation at Rupture and the Corresponding Time for 0.5 per cent Molybdenum Steel with Differently Spheroidized Microstructures.

The order of appearance of the brittle fracture with increasing time is I, S, and M for the variable of degree of spheroidization; N, then A for the heat-treatment variable; and equal times for the F and C grain-size variable. The delayed changes in elongation, in type of fracture, and in true slope of the rupture-stress curves give a questionable extrapolation over the 10,000 to 100,000-hr. period.

In long-time high-temperature service the steels in initial condition I should ultimately spheroidize the carbides to condition S. It would be expected that the rupture elongations would finally rise towards those obtained for the *S* and M conditions of the steel. The author's previous creep-test paper[2] discussed the theoretical time at temperature necessary for carbide spheroidization, applying an exponential formula of the diffusion form tested over the range of 1300 to 1100 F., which extrapolated to 1000 F. indicates the time to change condition I to condition S as 10,000 hr. for the normalized steel N and over 30,000 hr. for the annealed steel *A*. In Fig. 3, at 1000 F., only the elongation curves for the initial conditions of the steels, CNI, FNI, and FAI, show a slight tendency to rise towards the elongations for the spheroidized steels as the above hours are approached. If stresses nearer to design values for 1000 F. were used in a very long time test, would the rupture-elongations increase?

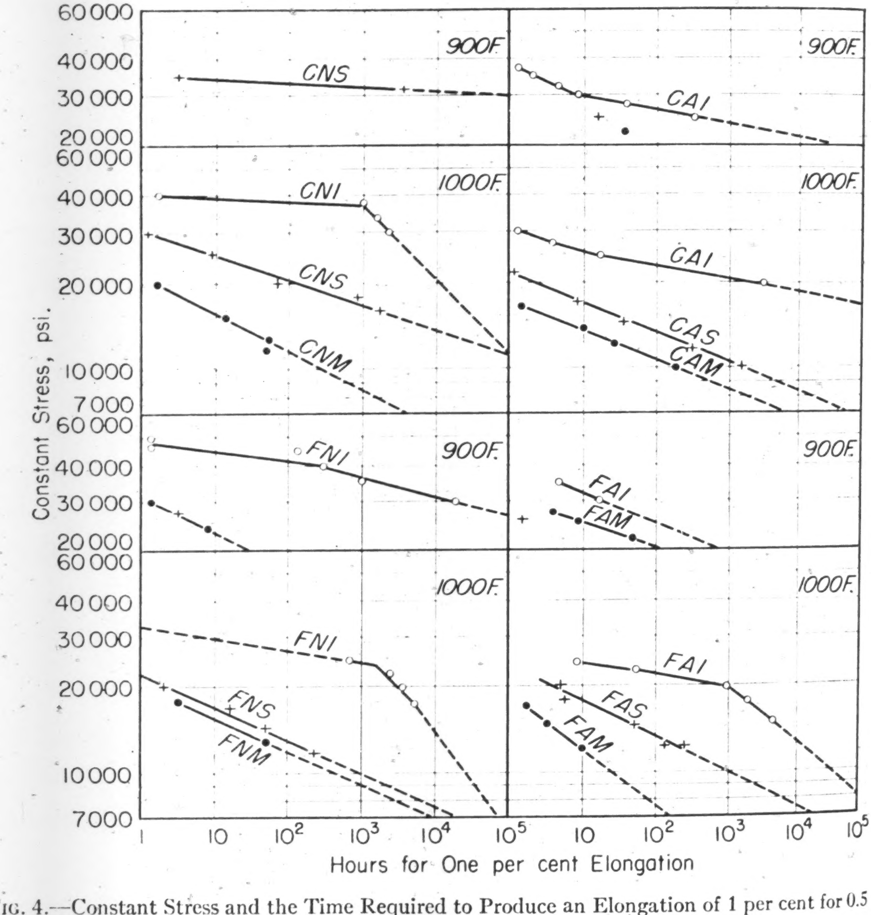

FIG. 4.—Constant Stress and the Time Required to Produce an Elongation of 1 per cent for 0.5 per cent Molybdenum Steel with Differently Spheroidized Microstructures.

Figure 4 illustrates the time required to produce 1 per cent elongation for the constant load stress applied to each specimen. Many of the individual tests do not appear on this plot because the stresses were above the yield point or the flow was greater than 1 per cent in the first hour. The data tabulated in Tables III and IV have many blank spaces due to the lack of test points or to questionable extrapolation.

Each of the longest time test specimens was especially examined for graphite formation in the steel, particularly near the fracture, and no graphite was observed.

TEST RESULTS

In Fig. 5 the rupture stress tested to 10,000 hr. is plotted against the time required to spheroidize the steel. The linear scale presents a perspective to the time required by the temperature to produce strength changes in the steel. Scales are added above the curves for 900 F. and below the figure for 1000 F. showing the equivalent spheroidizing time without stress at the rupture-stress test temperatures, using the formula discussed in the previous creep test paper[2] under the heading "Service Life." The previous approximation for service time at the rupture-test temperatures considered the test points at 55, 180, and 1500-hr. spheroidizing time at 1292 F. as equivalent to 7, 23, and 200 yr. in Fig. 5(*a*) and 0.7, 2.3 and 20 yr. in Fig. 5(*b*) for the service time at the temperature of the rupture tests.

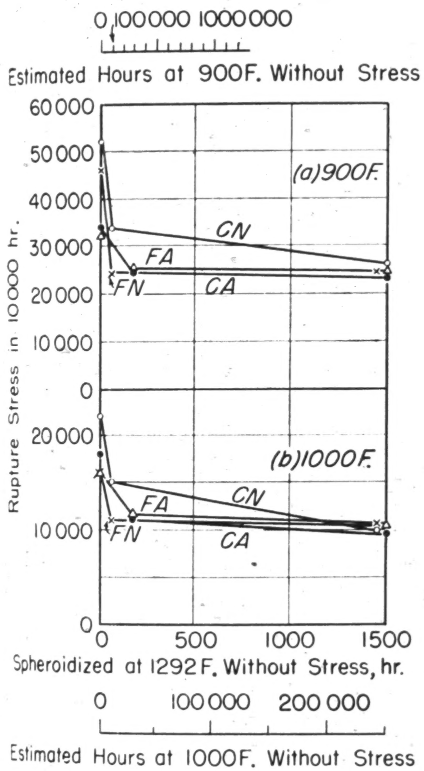

FIG. 5.—Rupture Stress for 10,000 hr. at 900 and 1000 F. for 0.5 per cent Molybdenum Steel when Spheroidized for Different Amounts of Time.

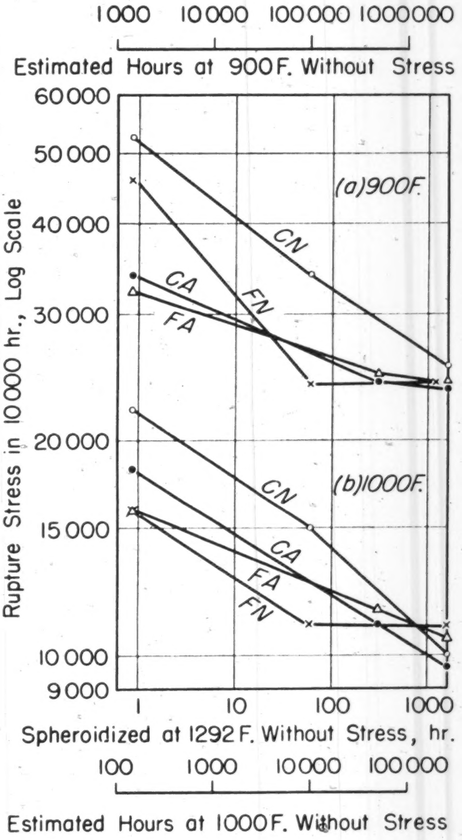

FIG. 6.—Log Plot of Rupture Stress at 900 and 1000 F. *Versus* Spheroidizing Time Without Stress for 0.5 per cent Molybdenum Steel.

Years	Hours (approx.)
0.7	6 100
2.3	20 000
7.0	61 000
20.0	174 000
23.0	200 000
200.0	1 740 000

The data indicate that during a service period of 23-yr. operation at 900 F. the rupture strength would decrease to between 78 and 52 per cent of the initial condition. The more rapid decrease in rupture strength occurs in the tests at 1000 F. where the data indicate that from 72 to 55 per cent of the initial stress is obtained at 2.3 yr. and from 66 to 45 per cent in 20 yr. of operation at 1000 F. The rupture-stress decrease with service time is less than that found in the creep tests on the same material as follows:

Service		Decrease in Strength, per cent	
Temperature, deg. Fahr.	Years (est.)	Rupture	Creep
900	23	78 to 52	72 to 46
1000	2.3	72 to 55	58 to 40
1000	20	66 to 45	41 to 23

Figure 6 presents the same data as Fig. 5 plotted on a log-log scale with the initial conditions placed at 0.9 hr. which is equivalent to the 4-hr. draw at 1200 F.

Figures 5 and 6 for the rupture stress show a striking parallel to the Figs. 15 and 16 previously presented[2] for the creep-stress tests on the same material. At a temperature of 900 F. the normalized structure N is the major factor in both the creep strength and rupture stress. But the fine-grained, normalized steel FN drops most rapidly, with respect to spheroidizing time, to the lowest values in all of the test series in both creep and rupture strength. When the normalized structure is combined with the coarse grain, CN, there is produced the strongest condition of the steel at 900 F. At a test temperature of 1000 F. the coarse grain is the predominating factor in both rupture and creep strength.

The results of the massed spheroid condition, M, show that the fine- and coarse-grained steels, in either normalized or annealed condition, appear with time at temperature to approach an approximately common value in the creep stress and another common value in the rupture stress.

SUMMARY

The results of the long-time rupture tests on 0.5 per cent molybdenum steel in different conditions of carbide spheroidization parallel the results of the creep tests made on the same material. While the initial rupture and creep stresses vary greatly for the different conditions of the steel, the data presented indicate that in approximately 20 yr. of operation some of the items would decrease in creep stress to 46 per cent and in rupture stress to 52 per cent at 900 F., and in creep stress to 23 per cent and in rupture stress to 45 per cent at 1000 F.

In the selection of heat-treatment for this steel the same conclusions apply to both the creep and rupture tests. At 900 F. the normalized structure is the major factor, and when combined with the coarse grain it produces the strongest steel. At 1000 F. the coarse grain predominates in both creep and rupture strength, and normalized or annealed structures are of minor importance.

DISCUSSION

Mr. R. F. Miller.[1]—In Fig. 3, Mr. Weaver shows that at 1000 F. the elongation curves for the initial conditions of steels CNI, FNI, and FAI show a slight tendency to rise toward the elongation of the spheroidized steels as the tests progress to long-time periods. The suggested explanation of this behavior is that the steels tend to spheroidize during test so their condition approaches that of the M or S conditions. It is estimated that the time to change condition I to condition S is 10,000 hr. for the normalized steel *N* and over 30,000 hr. for the annealed steel *A*.

In our study of the effect of strain rate on the strength and type of failure of carbon molybdenum steel,[2] we showed (Fig. 12) indications of the same behavior at 1100 F. where the changes take place somewhat more rapidly than at the temperatures of 900 and 1000 F. used by Mr. Weaver. It has been our experience that as the strain rate decreases (as the test time increases), the type of fracture changes from transgranular to intergranular, and the amount of elongation decreases. Mr. Weaver has found that the order of the appearance of the brittle fracture with the increase in time is I, S, and M. This is also confirmed by our experience at 1100 F. where the intergranular fracture appeared earlier in the pearlitic than in the spheroidized material.

Mr. Weaver is to be congratulated on the very thorough investigation which he has carried out on the effect of carbide spheroidization on the creep and rupture of carbon molybdenum steel. He has shown very clearly that carbide spheroidization may cause a decrease of strength of material in service at elevated temperature. Between one quarter and one half of the strength of carbon molybdenum steel would be lost during a service period of about twenty years at 900 F., and more than half of the strength would be lost in the same length of time at 1000 F. Up to the present time, surface stability (corrosion or scaling losses) and strength measurements made over a time period of 500 to 2000 hr. have served as the accepted basis for design. It now appears that the upper limiting temperature for usefulness of steels at elevated temperatures should be based upon their internal as well as their external stability and that adequate factors of safety must be employed if the service temperature is to exceed the upper limiting temperature for microstructural stability. The calculations which Mr. Weaver has made are a valuable contribution to the somewhat neglected subject of carbide spheroidization, and any further data which Mr. Weaver may have on this subject would be very much appreciated.

Mr. S. H. Weaver (*author's closure by letter*).—I wish to thank Mr. Miller for referring to the data which indicate a similar behavior occurring at 1100 F. but at a more rapid rate than that which we found at 900 and 1000 F. We agree with his recommendation that the changes in microstructure and properties of the steel under discussion are of sufficient magnitude to be considered in determining the factor of safety for apparatus requiring a long life at elevated temperatures.

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[1] Development Engineer, Stainless and Alloy Steels, Carnegie-Illinois Steel Corp., Pittsburgh, Pa.

[2] R. F. Miller, G. V. Smith, and G. L. Kehl, "Influence of Strain Rate on Strength and Type of Failure of Carbon Molybdenum Steel at 850, 1000, and 1100 F.," *Transactions*, Am. Soc. Metals, Vol. 32, pp. 817–848 (1943).

Since the preparation of this paper, additional test results have been obtained. As the stock of steel has been consumed during the past eight years, this series of tests has now been discontinued.

There was a question as to the accuracy of the empirical formula for the time to spheroidize the carbides when extrapolating from data obtained at higher temperatures to the lower temperatures of operation. This formula was discussed in the author's previous creep test paper[3] under the heading "Service Life."

Taylor[4] at the University of Michigan used an annealed, 0.13 per cent carbon, killed steel with test points at 1300 1250, and 1200 F., and drew a straight-line curve with the exponential constant *b* equal to 44 630, indicating 1900 hr. at 1100 F. With the average *b* value of 45 000, the time is 1970 hr. at 1100 F. His very accurate tests at 1100 F. gave 2100 hr. to fully spheroidize the carbides, or 7 per cent greater time than the formula.

The steel used in the present creep and rupture series was tested at the General Electric Laboratories in the normalized condition from 1292 to 1000 F. With *b* equal to 45 000 and 49 hr. at 1300 F., the equation indicates 55 hr. at 1292 F. and 9410 hr. at 1000 F.; the tests gave 55 and 10 000 hr. respectively, a 6 per cent longer time than the formula being required at 1000 F. to duplicate the microstructure of Fig. 9 in the creep paper.

Tests were made upon the effect of stress in decreasing the time to spheroidize the carbides fully. Taylor's tests on carbon steel at different constant stresses and temperature usually overspheroidized. The S.A.E. 1045 annealed steel gave an accurate result at 1250 F. and 1200 psi. in 150 hr., while zero stress required 228 hr. From these tests we made the assumption in estimating the service life under operating conditions, that the presence of stress reduced the time to spheroidize the carbides fully to two thirds of that found with no stress present.

Our tests on the carbon-molybdenum steel were made on eight tapered bars with the section at one end ten times that at the other end. At 1200 F. the annealed, tapered bar became fully spheroidized (CAS) in 496 hr. or two thirds of the time without stress, the 400-psi. end duplicating Fig. 12, while the 4000-psi. end had slightly larger spheroids with more clear space in the ferrite. The normalized bar became fully spheroidized (CNS) in 152 hr. or two thirds of the time without stress, the 4000-psi. end duplicating the condition of Fig. 9 "after creep at 900 F." while the 400-psi. end had larger spheroids and acicular structure. The remaining bars were in the furnace for longer periods and were overspheroidized. These data are not numerous enough to detail the influence of stress but give further evidence that working stresses reduce the time to spheroidize the carbides to approximately two thirds of the time required without stress.

The effect of temperature upon the time to spheroidize the carbides fully without stress may be seen from the multipliers in Table V calculated for 1 hr. at 1300 F. with *b* equal to 45,000. If the time t_1 is known for temperature

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[3] S. H. Weaver, "The Effect of Carbide Spheroidization Upon the Creep Strength of Carbon-Molybdenum Steel," Am. Soc. Testing Mats., Vol. 41, p. 608 (1941).

[4] J. H. Taylor, "A Study of Spheroidization in Steels at Temperatures Below the Critical Range," unpublished thesis, University of Michigan, August, 1934.

F_1, then at another temperature F_2, the required time t_2 is equal to t_1 divided by the multiplier under F_1, then multiplied by the multiplier under F_2. Table V, when plotted upon semi-log paper gives a straight line. Calculate $1/T$ at each F in the table, where T equals F plus 462. On the linear scale use $1/T$ values, marking at each tabular value the corresponding F temperature. Mark the multipliers on the log scale. All points from the table will be found to lie on one straight line.

The time to spheroidize the carbides without stress at 1300 F. ranges from 5 to 222 hr. depending upon the chemical composition and condition of the steel. The data accumulated so far are given in Table VI. So many factors enter into a determination of spheroidization time that it is difficult to draw any final conclusion. However, the information is presented in response to Mr. Miller's request.

TABLE V.—MULTIPLIERS FOR TIME AT DIFFERENT TEMPERATURES FULLY TO SPHEROIDIZE THE CARBIDES WITHOUT STRESS.[a]

Deg. Fahr.	1300	1250	1200	1150	1100	1050	1000	950	900
Multiplier	1.00	2.31	5.46	13.26	33.4	85.2	226.2	612	1772

[a] 1292 F. (700 C.) has multiplier 1.124.

TABLE VI.—TIME TO SPHERIODIZE THE CARBIDES WITHOUT STRESS AT 1300 F.

Item	Composition							Condition	Heat Treatment[a]	t_{13}[b]	*b*[c]	Tests by
	C	Si	Mn	Mo	V	Cr	Miscellaneous					
No. 1	0.13							Killed	Annealed	72	44 630	Mich.
No. 2									Normalized	22	42 200	
No. 3									Cold Worked	5	43 300	
No. 4	0.45						S.A.E. 1045	Wrought	Annealed	110	42 700	Mich.
No. 5	0.85						S.A.E. 1085	Wrought	Annealed	120	41 300	Mich.
No. 6	0.16	0.23	0.47	0.42				Wrought	Annealed	180	45 100	Mich.
No. 7	0.16	0.19	1.25	0.25				Wrought	Annealed	80	45 500	Mich.
No. 8	0.17	0.20	0.88	0.42			0.004 Al 0.021 Al_2O_3	Plate	1560F-8 hr.—F.C.	160	45 000	G.E.
No. 9									1560F-8 hr.—A.C.	49	45 000	G.E.
No. 10									1750F-8 hr.—F.C.	160	45 000	G.E.
No. 11									1750F-8 hr.—A.C.	49	45 000	G.E.
No. 12	0.18	0.25	0.62	0.55	0.12	0.07		Cast	1740F-8 hr.—A.C.	71		G.E.
No. 13	0.14			0.50				Pipe	1650F-8 hr.—F.C.	187		G.E.
No. 14	0.19	0.25	0.87	0.45			Si killed	Plate	1740F-8 hr.—F.C.	185		G.E.
No. 15									1740F-8 hr.—A.C.	63		G.E.
No. 16	0.33	0.34	0.22	1.09			Al—0.12	Casting	1700F-2 hr.—F.C.	91		G.E.
No. 17									1700F-2 hr.—A.C.	56		G.E.
No. 18	0.17	0.50	0.57	1.1	0.22			Casting	1900F-10 hr. Fast F.C.	22		G.E.
No. 19	0.14	0.48	0.66	1.10	0.14			Casting	1700F-2 hr.—F.C.	70		G.E.
No. 20									1700F-2 hr.—A.C.	26		G.E.
No. 21	0.14	0.18	0.42			5.12		Wrought	Annealed	150	43 600	Mich.
No. 22	0.15	0.25	0.42	0.51		5.18		Wrought	Annealed	222	43 600	Mich.
No. 23	0.07	0.72	0.42	0.54		1.25		Wrought	Annealed	70	45 300	Mich.
No. 24	0.12	0.14	0.45	0.54		0.45	1¼ lb. Al per ton	Pipe	1700F-2 hr.—F.C.	58		G.E.
No. 25									1700F-2 hr.—A.C.	22		G.E.
No. 26	0.13	0.22	0.50	0.56		0.48	Si-killed	Pipe	1700F-2 hr.—F.C.	135		G.E.
No. 27									1700F-2 hr.—A.C.	54		G.E.
No. 28	0.13	0.15	0.60	0.48		0.87		Pipe	As received	27		G.E.
No. 29	0.08	0.28	0.25			1.17	W-0.97	Wrought	Annealed	160	44 700	Mich.
No. 30	0.47	0.72	0.47		0.30	1.36	W-0.90	Wrought	Annealed	200	44 500	Mich.

[a] F.C. is furnace-cooled. A.C. is air-cooled or normalized.
[b] t_{13} is hours to fully spheroidize the carbides to condition S without stress at 1300 F.
[c] *b* is the constant in exponent of spheroidizing time equation.

RESISTANCE OF IRON-NICKEL-CHROMIUM ALLOYS TO CORROSION IN AIR AT 1600 TO 2200 F.*

By Anton deS. Brasunas,[1] James T. Gow,[1] and Oscar E. Harder[1]

A series of iron-nickel-chromium alloys has been studied for the Alloy Casting Institute with reference to their resistance to corrosion in air at temperatures of 1600, 1800, 2000, and 2200 F. Most of the cast-alloy materials used in this investigation were prepared by the Research Laboratory of the American Brake Shoe Co. The chromium variation has been from 11 to 36 per cent at 5 per cent intervals, and the nickel content was varied from zero to approximately 70 per cent at the 11 per cent chromium level and from zero to about 50 per cent at the 31 per cent chromium level. For the most part, the carbon content has been about 0.45 per cent, but higher and lower carbon contents have been introduced to study the effect of carbon on certain compositions. The silicon and manganese contents have approximated 1.25 and 0.75 per cent, respectively. However, silicon, too, has been varied in certain compositions to study its effect on corrosion resistance. There has been substantial variation in the nitrogen content, but this element was not intentionally varied, and no study has been made of its effect on corrosion resistance.

Most of the data are based on the results of tests run for a period of 100 hr. with the specimens being heated in a muffle furnace through which air was passed at the rate of 200 cu. cm. per minute. The air was saturated with water vapor at 90 F. and preheated so as not to affect the temperature of the specimens. Selected alloys have also been run in tests for 1000 hr. In one series they were heated continuously; in a second series they were cooled to room temperature at 24-hr. intervals to observe the effect of interrupted heating; and in a third series the specimens were intermittently cooled and tumbled to remove loose scale. The extent of corrosive attack has been determined by removing the corrosion products and determining the actual metal loss. The results are presented in the form of average corrosion rates expressed in "grams per square inch per day" and "inches penetration per year." The influence of metal-scale formation and flaking tendencies on rates of corrosion is briefly discussed.

While it is considered that this paper presents a good appraisal of the resistance of iron-nickel-chromium alloys to corrosion in air in the temperature range of 1600 to 2200 F., the authors wish to point out that the data may not apply when other atmospheres are used, and that data in this paper are not to be considered as directly usable in selecting alloy compositions for commercial installations, especially in cases where other types of atmospheres are involved. The data should serve principally as base-line information and as an indication of approximate alloy compositions

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] Research Engineer, Assistant Supervisor, and Assistant Director, respectively, Battelle Memorial Institute, Columbus, Ohio.

which may be suitable for high-temperature applications in certain atmospheres. Work is in progress for the Alloy Casting Inst. on other atmospheres, especially flue gases containing sulfur and of compositions representing different degrees of fuel combustion.

PREPARATION OF SPECIMENS

The alloys were melted in basic-lined induction furnaces and poured into dried sand molds. Wedge-shaped castings were produced, 1 in. thick at the bottom and $2\frac{3}{8}$ in. thick at the top, 6 in. in width, and 8 in. in height. The section 1 to 3 in. from the bottom was used for the corrosion specimens. After sectioning to provide suitable machining stock, specimens of 0.375-in. diameter by 1.000 in. long were machined to a ground finish. These were carefully checked with micrometers, and all variations were noted. All specimens were carefully inspected for porosity before being employed in the corrosion tests.

DESCRIPTION OF APPARATUS

An electrically heated muffle-type furnace was used to heat the specimens which were placed vertically, approximately $\frac{3}{4}$ in. apart, within the furnace chamber on a refractory holder. Temperature fluctuations were automatically held to ±10 F. by means of an electric controller actuated by a thermocouple placed within the furnace muffle, adjacent to the specimens. A second thermocouple was inserted periodically to assure positive temperature control, as determined by a portable potentiometer.

Ordinary air saturated with water vapor at 90 F. and preheated to furnace temperature was passed into the furnace chamber continually during the test.

TESTING PROCEDURE

When the specimens representing various alloy compositions were selected for testing at a particular temperature, they were treated as follows:

1. Specimens were measured with micrometers to determine their exact surface areas.
2. They were then degreased in carbon tetrachloride. From this point on, the specimens were handled with small tongs to eliminate possible surface contamination which might influence the rate of corrosion.
3. Each specimen was carefully weighed prior to exposure.
4. The specimens were then placed vertically on specially constructed refractory blocks which were inserted into the furnace chamber.
5. The desired furnace temperature was attained, and air, saturated with water vapor at 90 F. and preheated, was passed through the furnace at the rate of 200 cu. cm. per minute. These conditions were maintained during the entire test.
6. At the conclusion of the testing period, or at selected intervals, the specimens were allowed to cool to room temperature. During this cooling cycle, some specimens lost scale by vigorous flaking. Drafts were avoided during scaling, and in some of the tests, inverted crucibles were placed over the specimens in order to collect the scale thrown off.
7. After visual examination of the specimens to note the extent and adherence of the scale, the specimens were cathodically descaled in a molten caustic salt bath (60 per cent NaOH, 40 per cent Na_2CO_3)[2], using a current density of 400 amp. per square foot for a period of 1 to 4 min., depending on the nature of the scale. The specimens were then water-quenched from this salt bath.
8. To remove traces of the caustic, the specimens were then flash-dipped in

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[2] S. D. Heron, G. Calingaert, and F. J. Dykstra, "Electrolytic Descaling," *Journal*, Soc. Automotive Engrs., Vol. 37, December, 1935, p. 19.

a concentrated solution of HCl, saturated with arsenic trioxide (As_2O_3), and washed thoroughly with water.

9. After drying, the specimens were reweighed to determine the amount of metal lost by corrosion.

10. From the weight-change data and surface areas, the rate of metal loss was calculated in terms of grams per square inch per day, and by using an average value for the density of the iron-nickel-chromium alloys, the corrosion rate was also computed in terms of inches penetration per year.

100-HR. CORROSION TESTS

The compositions of the alloys studied, together with the corresponding metal-loss data at 1600, 1800, 2000, and 2200 F. as determined by 100-hr. tests, are summarized in Table I. It will be noted that, in general, the severity of corrosive attack is progressively reduced as the alloy content is increased, but is increased as the temperature is raised. However, to give a comprehensive picture of the resistance of these binary (Fe-Cr) and ternary (Fe-Ni-Cr) alloys, it is necessary to consider the influence of nickel and chromium separately and of these two metals simultaneously.

The Influence of Nickel on Air Corrosion:

The weight-loss data shown in Table I have been plotted on a logarithmic scale in Figs. 1 and 2, and the influence of nickel additions at various chromium levels is apparent. The beneficial influence of increased amounts of nickel is shown to be most prominent at the lower chromium levels. As the chromium content is raised, the influence of nickel becomes less pronounced. The following text discusses the influence of nickel at the different chromium levels.

11 per cent Chromium.—At the 11 per cent chromium level, nickel additions from zero to 20 per cent result in a small increase in corrosion resistance; nickel variations between 20 and 50 per cent effect a more substantial change in corrosion resistance; and nickel contents in excess of 50 per cent have but a small further influence, especially at the lower temperatures. The over-all effect in reducing corrosion at the 11 per cent chromium level by going from zero to 70 per cent nickel is to reduce the extent of corrosion to one two-hundredth. This was found to be fairly consistent at all temperatures studied.

16 per cent Chromium.—The curves of Fig. 1, showing the influence of nickel additions at 16 per cent chromium level, indicate a marked improvement in corrosion resistance when the nickel content is raised from zero to 40 per cent. Beyond this amount, no further improvement is noted at 1600 F., but a slight improvement was observed at 1800, 2000, and 2200 F. The corrosion rate at 1800, 2000, and 2200 F. of 16 per cent chromium alloys is reduced to one eightieth by increasing the nickel content from zero to 70 per cent. At 1600 F., the corrosion was less severe, and a reduction to one twenty-fifth in the corrosion rate was observed.

21 per cent Chromium.—As shown in Fig. 2, the presence of 21 per cent chromium reduced the corrosion of this series of alloys considerably as compared with the 16 per cent chromium series. At 1600 F., the rate of corrosion was only slightly affected by nickel variations of zero to 62 per cent. At 1800 F., a moderate improvement in corrosion was effected by nickel additions of zero to 20 per cent. Nickel additions beyond this amount, up to about 60 per cent, have a negligible effect on corrosion resistance. At 2000 F., the corrosion rates are more severe, and amounts of nickel up to 30 per cent reduce the degree of corrosion. Nickel in excess of this amount seems ineffective. At 2200 F., alloys containing 21 per cent chromium

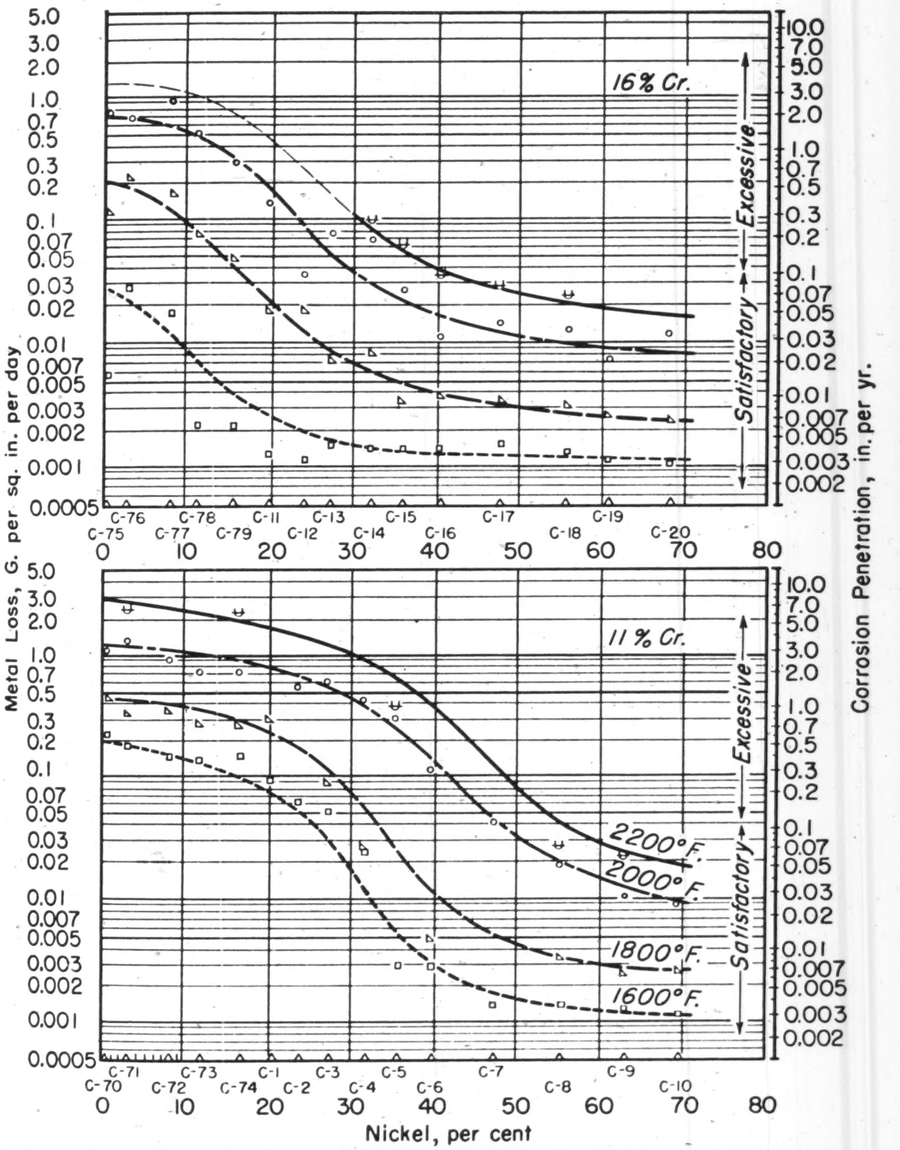

FIG. 1.—The Influence of Increasing Amounts of Nickel at the 11 and 16 per cent Chromium Levels on Air Corrosion of Alloys at 1600, 1800, 2000, and 2200 F., as determined in 100 hr. Tests.

appear to become more resistant to corrosion as the nickel content is increased from zero to about 20 per cent. Increasing the nickel content beyond about

TABLE I.—SUMMARY OF METAL-LOSS DATA OF IRON-NICKEL-CHROMIUM ALLOYS BY AIR CORROSION AT 1600, 1800, 2000, AND 2200 F.

Alloy	Composition of Alloys, per cent						Metal Loss by Corrosion							
							Grams per sq. in. per day				Inches Penetration per year			
	Carbon	Nickel	Chromium	Silicon	Manganese	Nitrogen	1600 F.	1800 F.	2000 F.	2200 F.	1600 F.	1800 F.	2000 F.	2200 F.
ZERO PER CENT CHROMIUM ALLOY														
No. C-99	0.85	0	0	0.2	0.4	....	0.4799	1.267	2.28	....	1.34	3.55	6.38	....
5 PER CENT CHROMIUM ALLOYS														
No. CA-11[b]	0.19	0	6.00	0.48	0.66	....	0.4451	0.7552	....	....	1.25	2.12	....	....
No. CA-12[b]	0.19	0	4.71	0.34	0.68	....	0.5497	0.7189	....	....	1.54	2.01	....	....
11 PER CENT CHROMIUM ALLOYS														
No. C-70	0.45	0.13	11.0	1.31	0.78	0.05	0.2291	0.4552	1.0380	....	0.641	1.27	2.91	....
No. C-71	0.45	3.0	10.9	1.30	0.76	0.05	0.1843	0.3315	1.3982	2.3795	0.516	0.928	3.92	6.67
No. C-72	0.45	8.3	11.0	1.28	0.75	0.06	0.1511	0.3441	0.9263	....	0.423	0.963	2.59	....
No. C-73	0.45	12.1	11.0	1.22	0.78	0.05	0.14	0.2873	0.7577	....	0.39	0.804	2.12	....
No. C-74	0.40	16.1	11.0	1.20	0.72	0.05	0.1593	0.2857	0.7512	2.2544	0.449	0.800	2.10	6.31
No. C-1	0.34	20.13	11.21	1.21	0.76	0.046	0.0925[a]	0.3055	....	....	0.259[a]	0.855	....	....
No. C-2	0.34	23.66	11.01	1.21	0.76	0.046	0.0612[a]	....	0.5680	....	0.171[a]	0.566	1.59	....
No. C-3	0.33	27.37	10.84	1.21	0.76	0.046	0.0522[a]	0.0897	0.62	....	0.146[a]	0.251	1.74	....
No. C-4	0.35	31.84	11.12	1.28	0.82	0.035	0.0245[a]	0.0262	0.4303[a]	....	0.069[a]	0.073	1.20[a]	....
No. C-5	0.35	35.57	11.06	1.28	0.82	0.035	0.0030	....	0.3033[a]	0.3564	0.008	....	0.849[a]	0.998
No. C-6	0.39	39.31	10.77	1.28	0.82	0.035	0.0028[a]	0.0047	0.1081[a]	....	0.008[a]	0.013	0.303[a]	....
No. C-7	0.31	47.34	11.09	1.28	0.84	0.022	0.0014	....	0.0415[a]	....	0.004	....	0.116[a]	....
No. C-8	0.37	55.09	11.21	1.28	0.84	0.022	0.0014[a]	0.0032	0.0191	0.0275	0.004[a]	0.009	0.053	0.077
No. C-9	0.32	63.03	10.98	1.36	0.90	0.008	0.0013[a]	0.0025	0.0103[a]	0.0224	0.004[a]	0.007	0.029[a]	0.063
No. C-10	0.37	69.07	10.87	1.36	0.90	0.008	0.0011[a]	0.0027	0.0088[a]	....	0.003[a]	0.008	0.025[a]	....
16 PER CENT CHROMIUM ALLOYS														
No. C-75	0.47	0.11	16.0	1.26	0.78	0.03	(0.0059)	(0.1238)	0.7610	...	(0.017)	(0.347)	2.13	....
No. C-76	0.43	3.0	16.1	1.23	0.75	0.04	0.0281	0.2360	0.7020	....	0.079	0.661	1.97	....
No. C-77	0.45	8.2	15.7	1.20	0.75	0.06	0.0180	0.1783	0.9374	....	0.050	0.499	2.63	....
No. C-78	0.46	12.0	15.8	1.22	0.77	0.06	0.0022	0.0793	0.5216	....	0.006	0.222	1.46	....
No. C-79	0.44	16.2	15.8	1.18	0.69	0.06	0.0022	0.0497	0.3031	....	0.006	0.139	0.849	....
No. C-11	0.47	20.00	16.17	1.16	0.80	0.050	0.0014[a]	0.0194	0.1406[a]	....	0.004[a]	0.054	0.394[a]	....
No. C-12	0.48	24.10	16.02	1.16	0.80	0.050	0.0012[a]	0.0195	0.0359[a]	....	0.003[a]	0.055	0.101[a]	....
No. C-13	0.50	27.55	16.25	1.16	0.80	0.050	0.0016[a]	0.0076	0.0803[a]	....	0.004[a]	0.021	0.225[a]	....
No. C-14	0.45	32.20	15.98	1.24	0.80	0.075	0.0015[a]	0.0087	0.0705[a]	0.0916	0.004[a]	0.024	0.197[a]	0.256
No. C-15	0.45	36.43	15.98	1.24	0.80	0.075	0.0015[a]	0.0036	0.0287[a]	0.0615	0.004[a]	0.010	0.080[a]	0.172
No. C-16	0.46	40.34	15.98	1.24	0.80	0.075	0.0015[a]	0.0040	0.0118[a]	0.0353	0.004[a]	0.011	0.033[a]	0.099
No. C-17	0.40	47.95	15.73	1.25	0.84	0.057	0.0017[a]	0.0037	0.0162[a]	0.0290	0.005[a]	0.010	0.045[a]	0.081
No. C-18	0.40	56.07	15.86	1.25	0.84	0.057	0.0014[a]	0.0034	0.0140[a]	0.0249	0.004[a]	0.010	0.039[a]	0.070
No. C-19	0.39	60.95	15.73	1.24	0.84	0.032	0.0012[a]	0.0028	0.0077[a]	....	0.004[a]	0.008	0.022[a]	....
No. C-20	0.42	68.13	15.90	1.24	0.84	0.032	0.0011[a]	0.0026	0.0130[a]	....	0.003[a]	0.007	0.036[a]	....
21 PER CENT CHROMIUM ALLOYS														
No. C-80	0.44	0.14	20.8	1.31	0.78	0.05	0.0026	(0.0052)	0.0348	0.1210	0.007	(0.015)	0.097	0.339
No. C-81	0.43	3.0	21.0	1.28	0.73	0.03	0.0040[a]	0.0292	0.0347	0.0544[a]	0.011[a]	0.082	0.097	0.152[a]
No. C-82	0.45	8.2	21.1	1.25	0.75	0.07	0.0019	0.0259	0.0333	0.0295[a]	0.005	0.073	0.093	0.083[a]
No. C-83	0.47	12.1	20.8	1.23	0.76	0.06	0.0015	0.0144	0.0274	0.0218[a]	0.004	0.040	0.077	0.061[a]
No. C-84	0.44	16.3	20.7	1.18	0.73	0.06	0.0021	0.0049	0.0285	0.0187[a]	0.006	0.014	0.080	0.052[a]
No. C-31	0.46	19.95	20.96	0.76	0.73	0.058	0.0035[a]	0.0055[a]	0.0182	....	0.010[a]	0.015[a]	0.051	...
No. C-32	0.46	24.08	20.92	0.76	0.73	0.058	0.0026[a]	0.0054[a]	0.0163	0.0672	0.007[a]	0.015[a]	0.046	0.188
No. C-33	0.47	28.34	20.85	0.76	0.73	0.058	0.0020	0.0052	0.0128	0.0576	0.006	0.015	0.036	0.161
No. C-34	0.47	32.32	20.92	1.21	0.77	0.072	0.0023	0.0040	0.0109	0.0719[a]	0.006	0.011	0.031	0.201[a]
No. C-35	0.47	36.02	21.03	1.31	0.77	0.064	0.0016	0.0036	0.0136	0.0502	0.004	0.010	0.038	0.141
No. C-36	0.46	40.08	20.98	1.29	0.76	0.064	0.0041[a]	0.0033	0.0144	0.0468[a]	0.011[a]	0.009	0.040	0.131[a]
No. C-37	0.46	47.90	21.00	1.27	0.77	0.080	0.0019[a]	0.0032	0.0101[a]	0.0274[a]	0.005[a]	0.009	0.028[a]	0.077[a]
No. C-38	0.45	55.92	20.77	1.35	0.81	0.067	0.0014	0.0035	0.0111	0.0301	0.004	0.010	0.031	0.084
No. C-39	0.45	61.84	21.22	1.17	0.78	0.054	0.0023	0.0032	0.0103	0.0206	0.006	0.009	0.029	0.058

TABLE I.—*Continued*

Alloy	Composition of Alloys, per cent						Metal Loss by Corrosion							
							Grams per sq. in. per day				Inches Penetration per year			
	Carbon	Nickel	Chromium	Silicon	Manganese	Nitrogen	1600 F.	1800 F.	2000 F.	2200 F.	1600 F.	1800 F	2000 F.	2200 F.
							26 PER CENT CHROMIUM ALLOYS							
No. C-85	0.45	0.16	25.8	1.34	0.78	0.04	0.0035^a	0.0204	0.0353	0.1135^a	0.010^a	0.057	0.099	0.318^a
No. C-86	0.43	3.0	25.9	1.32	0.73	0.03	0.0029	0.0262	0.0418	0.0598^a	0.008	0.073	0.117	0.167^a
No. C-87	0.45	8.2	25.8	1.30	0.75	0.07	0.0024	0.0084	0.0320	0.0215^a	0.007	0.024	0.090	0.060^a
No. C-88	0.47	12.1	25.6	1.23	0.77	0.05	0.0013	0.0061	0.0190	0.0198^a	0.004	0.017	0.053	0.055^a
No. C-89	0.45	16.0	25.8	1.15	0.74	0.06	0.0014	0.0036	0.0115	0.0188^a	0.004	0.010	0.032	0.053^a
No. C-41	0.46	20.12	25.47	1.12	0.68	0.046	0.0026^a	0.0058^a	0.0117	0.0183	0.007^a	0.016^a	0.033	0.051
No. C-42	0.47	23.48	26.00	1.23	0.73	0.126	0.0037^a	0.0038	0.0166^a	0.0594^a	0.010^a	0.011	0.046^a	0.166^a
No. C-43	0.47	27.40	25.78	1.22	0.75	0.139	(0.0101)	0.0042	0.0133	0.0521	(0.028)	0.012	0.037	0.146
No. C-44	0.45	31.80	25.64	1.22	0.72	0.074	0.0042	0.0051^a	0.0129	0.0658	0.012	0.014^a	0.036	0.184
No. C-45	0.49	35.94	25.92	1.22	0.74	0.093	....	0.0047	0.0137	0.0303	....	0.013	0.038	0.085
No. C-46	0.45	40.15	26.08	1.24	0.74	0.087	0.0043	0.0041^a	0.0115	0.0254	0.012	0.011^a	0.032	0.071
No. C-47	0.49	48.04	25.93	1.17	0.77	0.083	0.0025^a	0.0045^a	0.0120^a	0.0256	0.007^a	0.013^a	0.034^a	0.072
No. C-48	0.42	55.82	25.94	1.27	0.73	0.079	0.0031	0.0029	0.0113	0.0208	0.009	0.008	0.032	0.058
							31 PER CENT CHROMIUM ALLOYS							
No. C-90	0.47	0.20	30.9	1.30	0.77	0.05	0.0042	0.0186	0.0100	0.0379	0.012	0.052	0.028	0.106
No. C-91	0.44	3.0	30.9	1.22	0.72	0.04	0.0037^a	0.0126	0.0139	0.0493	0.010^a	0.035	0.039	0.138
No. C-92	0.43	8.2	31.0	1.28	0.77	0.07	0.0017	0.0043	0.0061	0.0471	0.005	0.012	0.017	0.132
No. C-93	0.44	12.1	30.8	1.24	0.77	0.06	0.0026	0.0038	0.0104	0.0337	0.007	0.011	0.029	0.094
No. C-94	0.46	15.9	30.8	1.19	0.73	0.06	0.0027	0.0037	0.0257	....	0.008	0.010	0.072	....
No. C-51	0.43	20.12	30.71	1.12	0.73	0.062	0.0063^a	0.0092	0.0142	0.0388	0.018^a	0.026	0.040	0.109
No. C-52	0.44	23.81	30.98	1.13	0.72	0.058	0.0050^a	0.0126	0.0120^a	0.0474	0.014^a	0.035	0.034^a	0.133
No. C-53	0.43	27.44	31.09	1.17	0.73	0.057	0.0076^a	0.0100	0.0219	0.0259	0.021^a	0.028	0.061	0.073
No. C-54	0.44	31.28	30.79	1.19	0.73	0.083	0.0081	0.0098	0.0131	0.0285	0.023	0.027	0.037	0.080
No. C-55	0.43	36.42	30.35	1.10	0.71	0.081	0.0046^a	0.0080	....	0.0305	0.013^a	0.022		0.085
No. C-56	0.48	40.25	30.68	1.18	0.70	0.114	0.0040^a	0.0080	0.0183^a	0.0184	0.011^a	0.022	0.051^a	0.052
No. C-57	0.44	48.35	30.62	1.25	0.67	0.093	0.0081	0.0083	0.0221	0.0199	0.023	0.023	0.062	0.056
							36 PER CENT CHROMIUM ALLOYS							
No. C-64	0.45	31.85	35.50	1.15	0.68	0.079	0.0139	0.0110	0.0152^a	0.0377	0.039	0.031	0.043^a	0.106
No. C-65	0.45	36.18	35.40	1.26	0.69	0.114	0.0072	0.0082	0.0299	0.0391	0.020	0.023	0.084	0.109
No. C-66	0.45	39.80	35.68	1.29	0.70	0.091	0.0067^a	0.0095	0.0121	....	0.019^a	0.027	0.034	....
							CONTROL SPECIMENS							
No. C-22 HT	0.48	35.50	16.15	1.33	0.80	0.069	0.0021	0.0056	0.0156^a	0.0485^a	0.006	0.016	0.044^a	0.136^a
No. C-21 HH	0.33	11.45	26.01	0.56	0.49	0.122	0.0050	0.0096^a	0.0181^a	0.0191	0.014	0.027^a	0.051^a	0.053
No. C-26 HH	0.44	11.58	26.20	1.28	0.68	0.073	0.0011	0.0143	0.0130	0.0155^a	0.003	0.040	0.036	0.043^a

[a] Averages of two or more tests.
[b] Alloys CA-11 and CA-12 also contain 0.56 per cent molybdenum.

20, up to 30 per cent, results in a slight increase in corrosion, but additions beyond 30 per cent tend to improve the alloy moderately.

26 per cent Chromium.—The 26 per cent chromium alloys were found to have corrosion tendencies quite similar to those observed at the 21 per cent chromium level. That is, sufficient chromium was present to render the alloys quite corrosion resistant, and the nickel content of the alloy had but minor or negligible influence. However, at 2000 F., there is a trend for the corrosion to decrease up to about 15 per cent nickel and to increase slightly in the range of about 20 to 40 per cent nickel.

31 per cent Chromium.—At the 31 per cent chromium level, the corrosion resistance of the variable nickel alloys

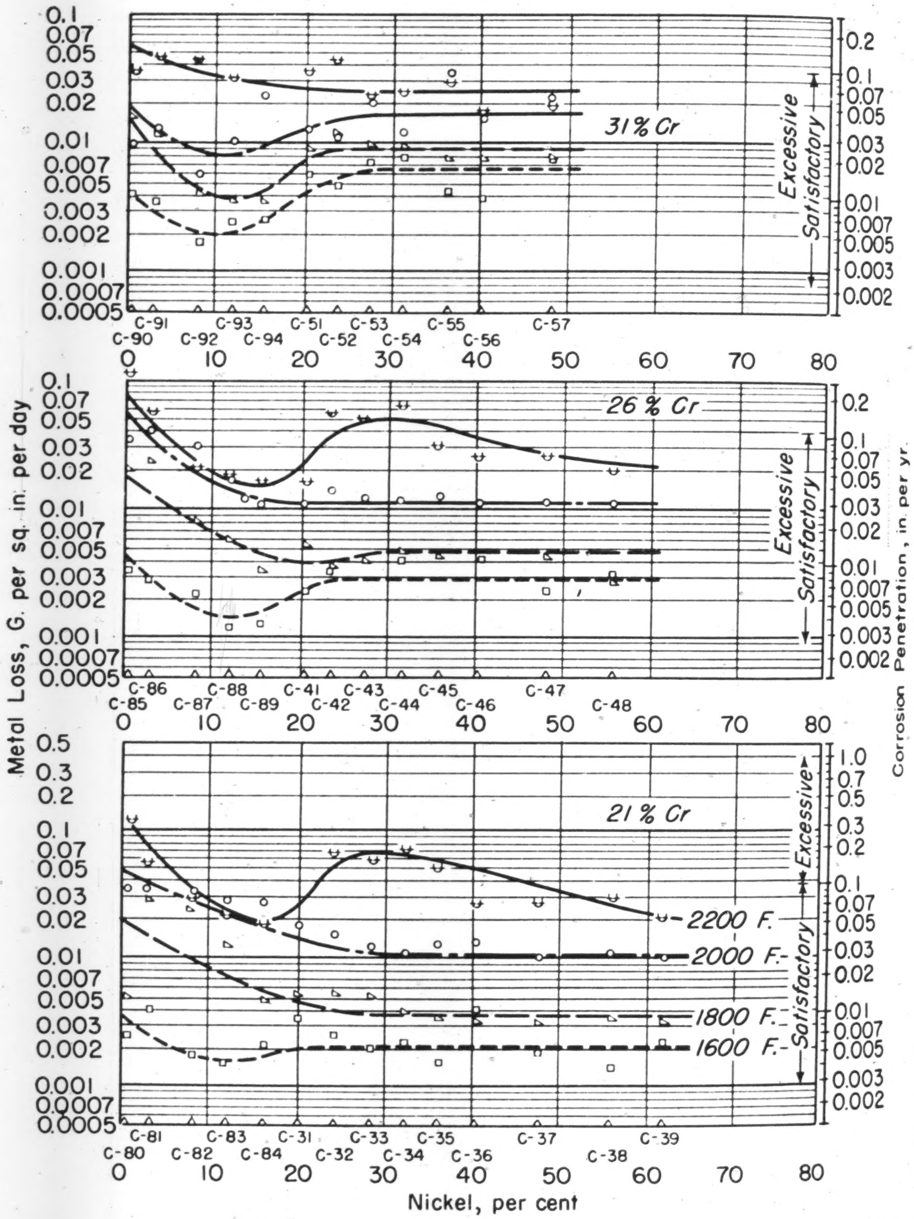

FIG. 2.—The Influence of Increasing Amounts of Nickel at the 21, 26, and 31 per cent Chromium Levels on Air Corrosion of Alloys at 1600, 1800, 2000, and 2200 F., as Determined in 100-hr. Tests.

appears to be similar to that of the corresponding alloys at the 26 and 21 per cent chromium levels. That is, nickel is not essential in imparting corrosion resistance to these alloys, because at these chromium levels the iron-chromium alloys are highly resistant to corrosion in air.

Nickel from 10 to 20 per cent appears to be optimum at the chromium levels of 21, 26, and 31 per cent. Nickel contents above and below this range have generally resulted in slightly greater corrosion. This optimum nickel range appears to shift, and the extent of improvement in corrosion resistance exhibited varies somewhat, depending upon the conditions of exposure. The increase in corrosion with increase in nickel in certain ranges is not completely understood but is thought to be real and is probably related to the rate and type of scale formed.

36 per cent Chromium.—The influence of nickel on corrosion resistance could not be ascertained at this chromium level because of the limited range of alloy composition available. However, there appears to be an indication that the 36 per cent chromium alloys are similar to the 31 per cent chromium alloys, but possibly slightly more severely attacked. Curves are not shown for this chromium level, but data are given in Table I.

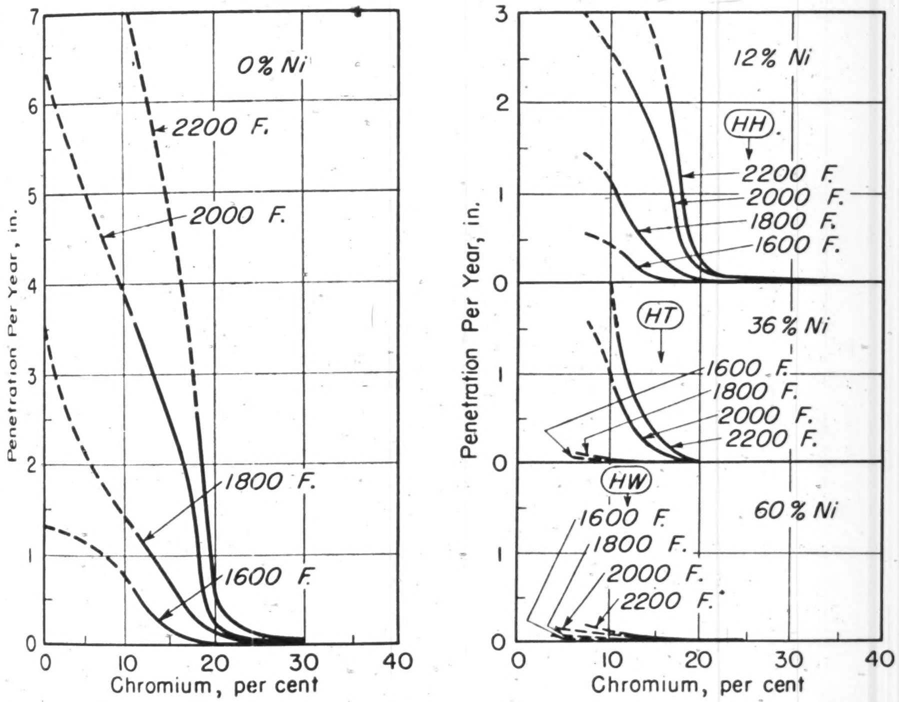

Fig. 3.—The Effect of Chromium at Various Nickel Levels on Air Oxidation of Alloys at 1600, 1800, 2000, and 2200 F.

The Influence of Chromium on Air Corrosion:

Figure 3 illustrates the influence of chromium at several nickel levels on air corrosion of alloys at 1600, 1800, 2000, and 2200 F. These curves have been drawn between 11 and 31 per cent chromium, the composition limits of the alloys studied. The broken portions of the curves at 0 per cent nickel for alloys containing less than 11 per cent chromium were approximately established by two materials: one a carbon steel (C-99), and the other a 5 per cent chromium steel (CA-11 and CA-12). The broken lines at the 12, 36, and 60 per cent nickel levels are extrapolations from the available data. The nickel levels of 12, 36, and 60 per cent were selected for the graphs shown in Fig. 3 because of extensive commercial use of alloys of these nickel contents. It is to be noted that the log scale in Figs. 1 and 2 has not been used in Fig. 3. This was because the data available were not sufficiently extensive.

It is quite apparent from Fig. 3 that, with no nickel present, chromium additions up to 20 per cent are exceptionally influential in reducing the extent of metal loss by corrosion, especially at the higher temperatures. Chromium additions beyond 20 per cent are shown to effect only small changes in corrosion resistance. However, at 2200 F., amounts up to 25 per cent chromium are shown to be beneficial.

At the 12 per cent nickel level, the effect of chromium is strong, though noticeably less than at zero per cent nickel. At 1600 and 1800 F., 20 per cent chromium, with the 12 per cent nickel, appears to be sufficient to reduce the corrosion rate to a minimum. At higher temperatures, however, greater amounts of chromium appear to be advantageous.

Alloys containing 36 per cent nickel are indicated to have about equally good corrosion resistance at 1600 and 1800 F. when 10, 20, or 30 per cent chromium is present. However, at 2000 and 2200 F., alloys containing less than 20 per cent chromium were noted to be quite susceptible to air corrosion. Twenty per cent chromium alloys were indicated to have good corrosion-resistant properties, and increasing the chromium content beyond this amount resulted in little or no further reduction in corrosion.

At the 60 per cent nickel level, alloys containing 11 per cent chromium were noted to have good corrosion resistance at 1600 and 1800 F., but at 2000 and 2200 F., at least 14 per cent chromium is indicated to be required for optimum corrosion resistance.

The symbols HH, HT, and HW which appear in Fig. 3 and other diagrams are the Alloy Casting Inst. designations for the 25 per cent chromium, 12 per cent nickel; 36 nickel, 16 chromium; and 60 nickel, 12 chromium alloys, respectively.

The Influence of Chromium and Nickel on Air Corrosion:

The contour curves on the ternary diagrams of Figs. 4, 5, 6, and 7 indicate the combined effects of nickel and chromium in imparting corrosion resistance, as determined by 100-hr. tests. The actual metal losses expressed in inches penetration per year are placed on the ternary diagrams at the locations which correspond to the actual composition of the specimens tested. Isocorrosion lines which represent different fixed intensities of corrosion, arbitrarily selected, were drawn through the points which represent those compositions that tend to corrode to the same extent.

At 1600 F.—The influence of composition on the extent of corrosion of iron-nickel-chromium alloys at 1600 F. is illustrated in Fig. 4. The rates of corrosion penetration range from 0.641 to 0.003 in. per year, depending upon the composition of the alloy. Nickel and chromium both are shown to reduce the corrosion rate. Chromium is considerably more effective than nickel, as is indicated by the slopes of the isocorrosion lines. The relationship between

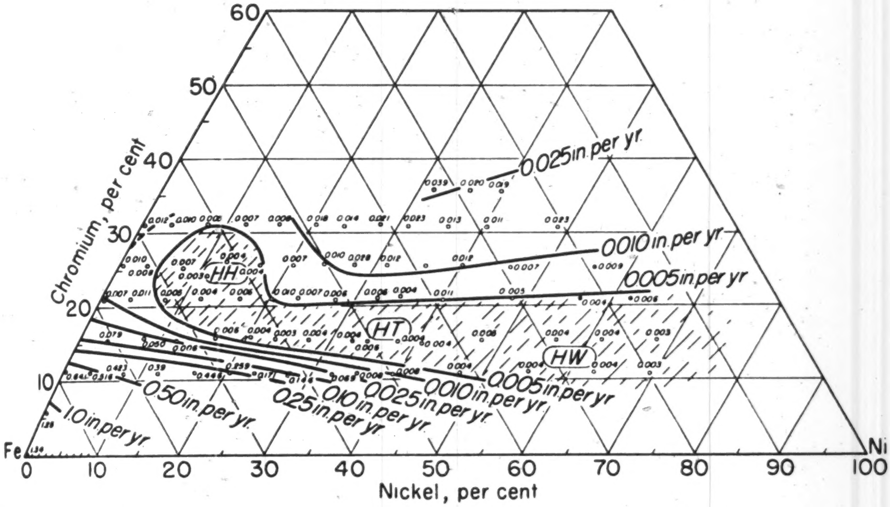

FIG. 4.—Metal Loss in Air Atmosphere at 1600 F. With Corrosion Expressed in Inches Penetration per Year. Alloys also contain about 0.4 per cent carbon, 1.25 per cent silicon, 0.75 per cent manganese, balance iron. Above data are based on 100-hr. tests.

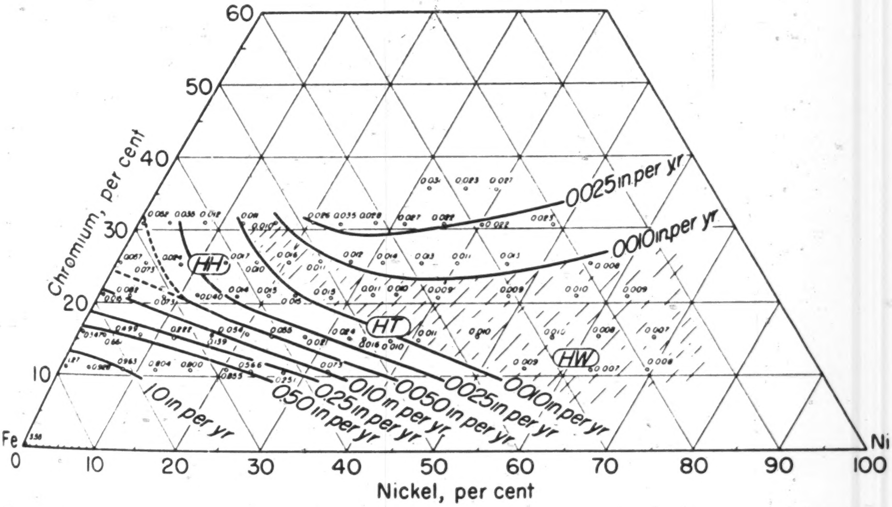

FIG. 5.—Metal Loss in Air Atmosphere at 1800 F. With Corrosion Expressed in Inches Penetration per Year. Alloys also contain about 0.4 per cent carbon, 1.25 per cent silicon, 0.75 per cent manganese, balance iron. Above data are based on 100-hr. tests.

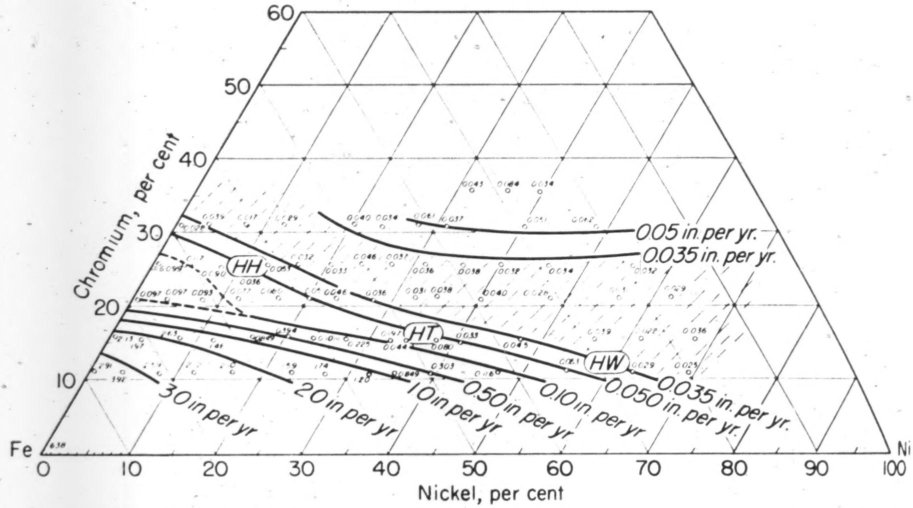

FIG. 6.—Metal Loss in Air Atmosphere at 2000 F. With Corrosion Expressed in Inches Penetration per Year. Alloys also contain about 0.4 per cent carbon, 1.25 per cent silicon, 0.75 per cent manganese, balance iron. Above data are based on 100-hr. tests.

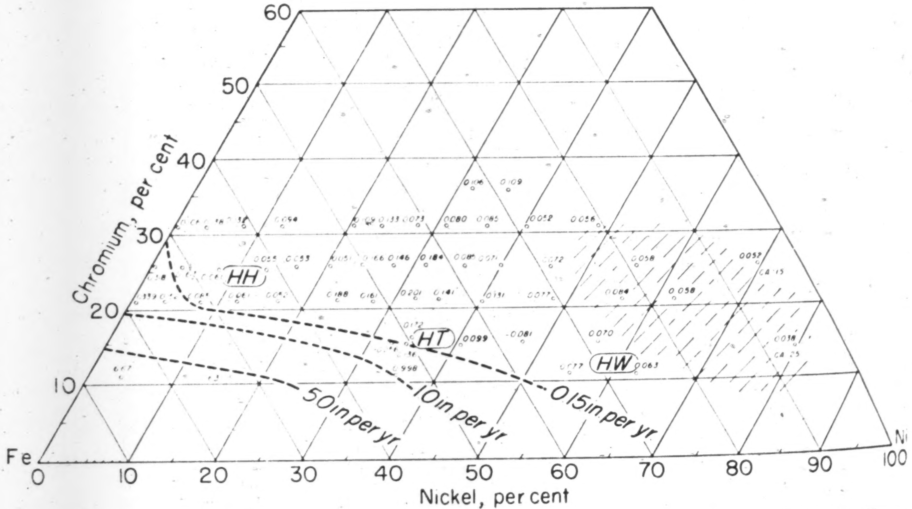

FIG. 7.—Metal Loss in Air Atmosphere at 2200 F. With Corrosion Expressed in Inches Penetration per Year. Alloys also contain about 0.4 per cent carbon, 1.25 per cent silicon, 0.75 per cent manganese, balance iron. Above data are based on 100-hr. tests.

the effectiveness of nickel and chromium is not constant, but is altered as the chromium and/or nickel content is varied.

There is a region of nickel and chromium contents which shows optimum corrosion resistance for this set of conditions, above and below which the corrosion rate is increased. This area has been cross-hatched, and it will be noted that three well-known commercial alloys[3] are in this area. The zone of optimum corrosion resistance has been observed to vary somewhat, depending upon the temperature and time of exposure, but such plateaus of good corrosion resistance have been noted at all of the temperatures studied.

At 1800 F.—Figure 5 is a similar graphic representation showing the extent of air corrosion at 1800 F. At this temperature, the range in corrosion penetration of the alloys is from 1.27 to 0.007 in. per year. The relative influences of nickel and/or chromium are similar to those noted at 1600 F. However, there is a definite indication of a shift toward alloys of higher chromium content to provide a given resistance to corrosion. This indicates a need for greater amounts of chromium at the higher exposure temperatures, but it is to be noted that increasing the nickel improves the corrosion resistance, especially up to about 20 per cent chromium.

At 2000 F.—The ternary diagram of Fig. 6 indicates that at 2000 F. the zone of optimum corrosion resistance has shifted to even higher alloy contents than at 1800 F. The range in corrosion penetration at this temperature is indicated to be from 3.92 to 0.022 in. per year. Between 20 and 30 per cent chromium at the low-nickel area, there appears to be a plateau where the corrosion rate is constant over a moderate range of chromium variations. This has been observed at all of the temperatures studied.

At 2200 F.—The metal-loss data of the alloys exposed to air corrosion at 2200 F. are presented in Fig. 7. The range in corrosion penetration is noted to vary from 6.67 to 0.055 in. per year. With two exceptions, the lower alloy metals have not been investigated at 2200 F., since their life expectancy is so short that their use at this temperature would be impractical. Since only the better alloys were investigated, only a small variation in the corrosion rates was found, and the composition limits for certain intensities of corrosion could not be established with certainty. To indicate this uncertainty, the isocorrosion lines were drawn broken.

TABLE II.—THE MINIMUM AMOUNTS OF NICKEL REQUIRED AT VARIOUS CHROMIUM LEVELS FOR SATISFACTORY CORROSION RESISTANCE.[a]

Chromium Content, per cent	Minimum Nickel Content, per cent			
	1600 F.	1800 F.	2000 F.	2200 F.
11........	26	34	48	56
16........	0	16	30	41
21........	0	0	4	9[b]
26........	0	0	3	5[c]
31........	0	0	0	8

[a] A corrosion penetration rate of 0.1 in. per year is arbitrarily selected to differentiate between satisfactory and unsatisfactory corrosion resistance.

[b] Corrosion resistance somewhat inferior from about 20 to 50 per cent nickel.

[c] Corrosion resistance somewhat inferior from about 20 to 40 per cent nickel.

TABLE III.—THE MINIMUM AMOUNTS OF CHROMIUM REQUIRED AT VARIOUS NICKEL LEVELS FOR SATISFACTORY CORROSION RESISTANCE.[a]

Nickel Content, per cent	Minimum Chromium Content, per cent			
	1600 F.	1800 F.	2000 F.	2200 F.
0........	16	22	23	31
10........	15	18	20	23
20........	13	15	18	21
30........	10	12	16	26
40........	10[b]	10[b]	13	22
50........	10[b]	10[b]	11	15
60........	10[b]	10[b]	10[b]	10
70........	10[b]	10[b]	10[b]	10[b]

[a] A corrosion penetration rate of 0.1 in. per year is arbitrarily selected to differentiate between satisfactory and unsatisfactory corrosion resistance.

[b] The slopes of the contour curves for these alloys indicate that even less than 10 per cent chromium will be satisfactory.

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[3] Designations and compositions were given earlier in this paper.

The Influence of Temperature:

If an arbitrary value such as 0.04 gram per square inch per day (approximately 0.1-in. penetration per year) is selected to differentiate between alloys suited for a particular service application and those whose corrosion rates are excessive, the data in Tables II and III illustrate the indicated minimum amounts of nickel and chromium required.

These tables indicate a need for greater alloy content as the exposure temperature is increased. Chromium is noticeably more efficient than nickel in providing corrosion resistance. There appear to be two general composition ranges where optimum corrosion resistance is attained; namely, in the vicinity of 26 per cent chromium, 20 per cent nickel and in the vicinity of 16 chromium, 60 nickel.

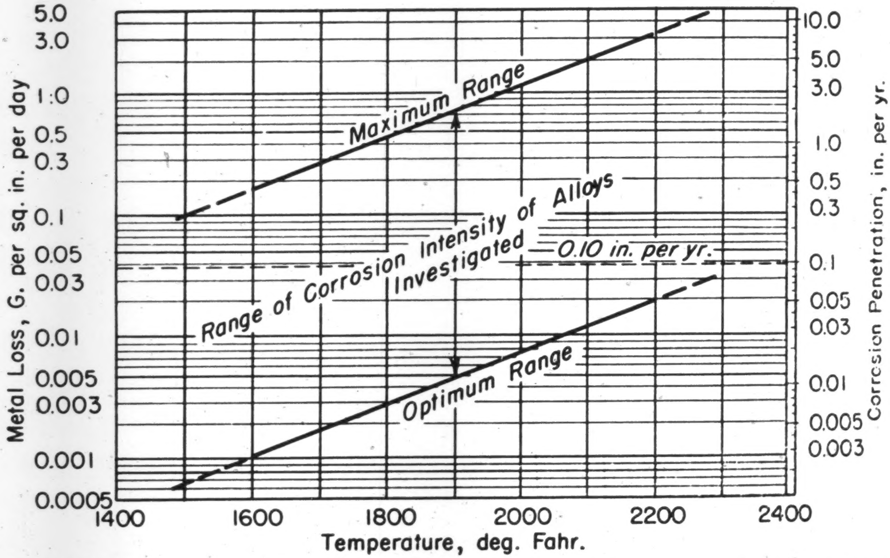

FIG. 8.—The Maximum and Minimum Intensities of Corrosion Encountered in Air Corrosion Tests of Iron-Nickel-Chromium Alloys at 1600, 1800, 2000, and 2200 F.

Increasing the exposure temperature from 1600 to 2200 F. at 200 F. increments results in increased corrosion for the entire alloy range in the approximate ratios of 1 to 2.7 to 7.0 to 20.0, or an increase in corrosion rate of 2.7 times for each 200 F. rise in temperature. This is illustrated in Fig. 8.

The optimum range shown in Fig. 8 indicates the minimum corrosion rate of the iron-nickel-chromium alloy range encountered at the various temperatures of testing. The alloys which show maximum corrosion at the various temperatures are not likely to be recommended for these temperatures by producers. Referring again to the arbitrarily selected value of about 0.1-in. penetration per year as acceptable corrosion resistance, it is evident from Fig. 8 that such a value is easily obtained at the lower temperature, but at 2200 F. optimum compositions are required.

Investigation of Miscellaneous Alloys:

Two special series of alloys were investigated to supplement the survey made on the corrosion resistance of iron-nickel-chromium alloys at elevated temperatures. These include a variable-carbon series, and a variable-silicon series of the 26 per cent chromium, 20 per cent nickel type alloy.

The Influence of Carbon on Air Corrosion:

Four alloys of the 26 per cent chromium, 20 per cent nickel type (C-95, C-96, C-97, and C-98) having carbon as a variable constituent were exposed to air corrosion at 1600, 1800, 2000, and 2200 F. Their actual compositions and calculated corrosion rates are presented in Table IV.

TABLE IV.—EFFECT OF CARBON AND SILICON VARIATIONS IN 26 CHROMIUM-20 NICKEL TYPE ALLOYS ON CORROSION RATES IN AIR AT 1600 TO 2000 F.

Alloy Number	Chemical Composition, per cent					Metal Loss							
						Grams per sq. in. per day				Inches Penetration per yea			
	Carbon	Nickel	Chromium	Silicon	Manganese	1600 F.	1800 F.	2000 F.	2200 F.	1600 F.	1800 F.	2000 F.	2200 F.
ALLOYS WITH CARBON VARIATIONS													
No. C-95	0.37	20.0	26.7	1.27	0.99	0.0014	0.0032	0.0111	0.0341	0.004	0.009	0.031	0.095
No. C-96	0.47	20.0	26.8	1.25	1.03	0.0009	0.0035	0.0112	0.0384	0.003	0.010	0.031	0.108
No. C-97	0.71	19.9	26.6	1.17	0.99	0.0018	0.0035	0.0111	0.0328	0.005	0.010	0.031	0.092
No. C-98	1.01	20.2	26.5	1.25	1.02	0.0026	0.0058	0.0129	0.1259	0.006	0.016	0.036	0.353
ALLOYS WITH SILICON VARIATIONS													
No. CA-1	0.32	19.9	26.0	0.50	0.50	0.0050	0.0040	0.0136	0.0297	0.014	0.012	0.038	0.083
No. CA-2	0.30	20.0	26.5	0.84	0.53	0.0037	0.0039	0.0117	0.0197	0.010	0.011	0.033	0.055
No. CA-3	0.30	19.7	26.3	1.21	0.53	....	0.0035	0.0093	0.0178	....	0.010	0.026	0.050
No. CA-4	0.30	20.1	26.7	1.62	0.58	0.0016	0.0020	0.0097	0.0175	0.004	0.006	0.027	0.049
No. CA-5	0.31	20.1	26.6	2.12	0.58	....	0.0022	0.0096	0.0155	....	0.006	0.027	0.043
No. CA-6	0.30	19.9	26.5	2.51	0.59	0.0013	0.0017	0.0052	0.0152	0.004	0.005	0.015	0.043
No. CA-7	0.30	19.8	26.6	3.08	0.60	0.0011	0.0014	0.0082	0.0171	0.003	0.004	0.023	0.048

The range in carbon content from 0.37 to 1.01 per cent for the alloys shown in Table IV is sufficient to make a substantial difference in the amount of chromium required for the formation of carbides and, therefore, in the amount of chromium remaining in the matrix. Assuming that all of the carbon combines with 16 times its weight of chromium, the calculated chromium content in the matrix of the low-carbon alloy will be about 20 per cent, whereas it will be only about 10 per cent in the alloy of highest carbon content. Thus, the ratio of chromium in the matrix of these two alloys will be about 2 to 1. Examination of the data in Table IV shows a trend for the corrosion to be more severe as the carbon content increases, but this variation is slight except in the tests at 2200 F., where the change in carbon content from 0.71 to 1.01 per cent increases corrosion three to four times. Certainly the test at 1600, 1800, and 2000 F. do not show the marked change in corrosion with increased carbon content which would be predicted from decreasing the chromium in the matrix from about 20 to about 10 per cent. This probably means that the major mechanism by which the chromium provides air-corrosion resistance in this type of alloy is by the formation of a protective scale, and that there is no essential difference in the ability of the scale to provide corrosion protection if the chromium in the scale was derived from chromium in the matrix or chromium in the form of the carbide. From the data now available, it appears that, for this type of alloy, variations in carbon contents within the range of 0.37 to 0.71 per cent have little effect on corrosion at temperatures in the range of 1600 to 2200 F., but that a further

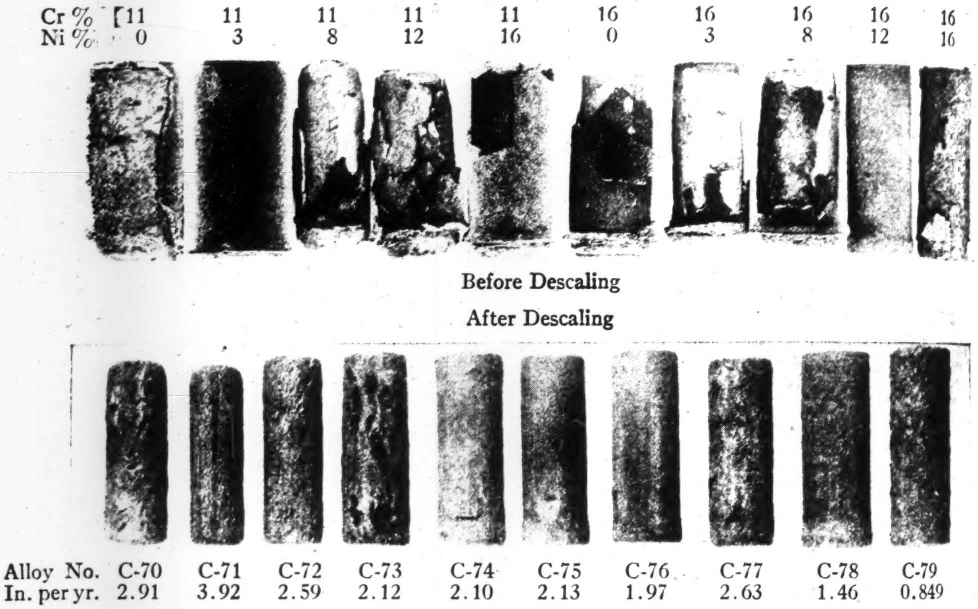
Cr % 11 11 11 11 11 16 16 16 16 16
Ni % 0 3 8 12 16 0 3 8 12 16
Before Descaling
After Descaling
Alloy No. C-70 C-71 C-72 C-73 C-74 C-75 C-76 C-77 C-78 C-79
In. per yr. 2.91 3.92 2.59 2.12 2.10 2.13 1.97 2.63 1.46 0.849

FIG. 9.—Appearance of Some Heavily Attacked Alloys Exposed to an Air Atmosphere at 2000 F. for 100 hr.

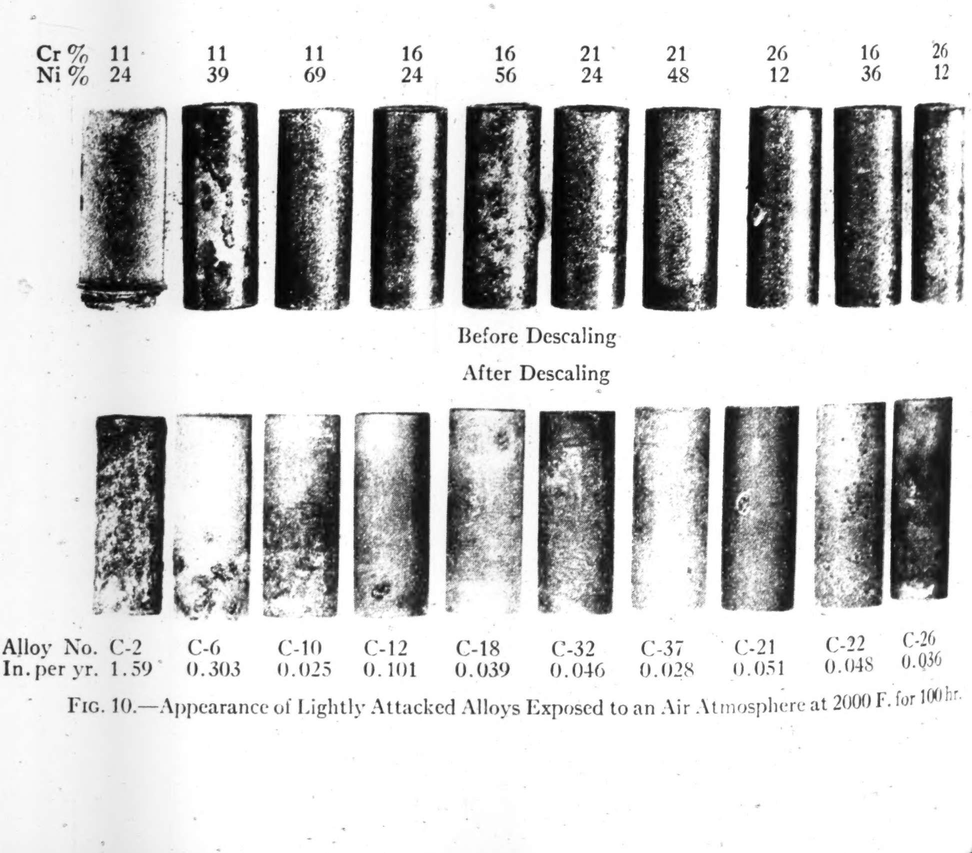
Cr % 11 11 11 16 16 21 21 26 16 26
Ni % 24 39 69 24 56 24 48 12 36 12
Before Descaling
After Descaling
Alloy No. C-2 C-6 C-10 C-12 C-18 C-32 C-37 C-21 C-22 C-26
In. per yr. 1.59 0.303 0.025 0.101 0.039 0.046 0.028 0.051 0.048 0.036

FIG. 10.—Appearance of Lightly Attacked Alloys Exposed to an Air Atmosphere at 2000 F. for 100 hr.

increase of 0.30 per cent carbon does make a substantial difference at 2200 F.

The Effect of Silicon on Air Corrosion:

The effect of variations in silicon content from 0.5 to 3.1 per cent in an alloy of the 26 per cent chromium, 20 per cent nickel type has been studied, and the data are also given in Table IV. Examination of these data shows that there is a trend for the corrosion to decrease as the silicon content is increased, particularly in the range of 0.5 to 1.5 or 2 per cent. This relation is found at all of the temperatures, and, in general, the lowest corrosion was found for silicon contents of 2.5 or 3 per cent. It must be pointed out, however, that alloys containing 26 per cent of chromium have good corrosion resistance in air and that the improvement by increasing the silicon content is not striking. It is recognized that, in alloys which do not have an adequate content of chromium to provide corrosion resistance, silicon is highly effective as a supplementary alloying element, as was pointed out by Harder and Gow[4] in connection with their work on an emergency heat-resistant alloy in which relatively low chromium contents were used, such as 10 to 12 per cent.

Appearance of Specimens After Corrosion Testing:

The examination of the specimens upon removal from the furnace indicated the formation of adherent, nonadherent, and various stages of semiadherent scales of the mono- and multilayer varieties. Figures 9 and 10 are photographs showing some of the variations in oxide scale formation of a few of the specimens which had been exposed to an air atmosphere at 2000 F. for a 100-hr. period. These photographs show the specimens before and after the electrolytic descaling operation. The nickel and chromium contents are listed above the individual specimens, and the calculated corrosion rates in terms of inches penetration per year are listed below them.

The influence of nickel and chromium content on the extent of corrosion is apparent in these photographs.

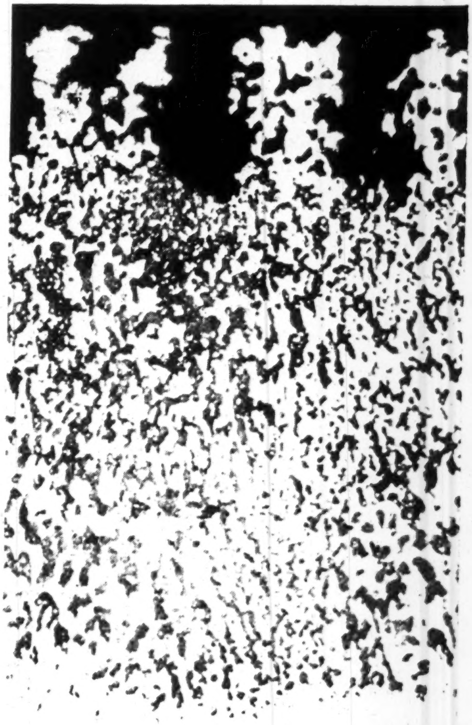

FIG. 11.—Semiscale Zone of Alloy C-72 After 100-hr. Exposure to Air Atmosphere at 2000 F. (×1000). Unetched.

Alloys containing small amounts of nickel (see Alloys C-71 and C-77 in Fig. 9) have been observed to be more strongly attacked than nickel-free or higher nickel alloys of the same chromium content. This is thought to be related in some way to the nature of the scale formed. The oxide scale of specimen C-71 in Fig. 9 is shown to be intact, whereas alloy C-78 appears to have had its scale flake off. The scale of alloy C-75, shown to be semiadherent, appears to be composed of four layers.

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[4] *Transactions*, Am. Soc. Metals, Vol. 32, p. 408 (1944).

The complete removal of the scale is essential to precision, since the corrosion rates are calculated from weight-loss determinations. However, this is not always possible. Alloys C-70, C-71, C-72, and C-73, when descaled (see Fig. 9), do not seem to have a completely metallic surface even after repeated and prolonged descaling. Upon microexamination, a semimetallic zone as shown in Fig. 11 was observed. This formation made complete descaling practically impossible so that a source of error was necessarily introduced with some compositions. However, this formation is associated with heavily attacked specimens, thereby making the percentage of error small. The data for descaled specimens are considered much more reliable than weight-gain and weight-loss data for scaled specimens which have been used in some published researches.

Specimens of higher nickel and chromium contents are shown in Fig. 10. The only specimen which shows excessive corrosion is C-2 with 24 per cent nickel and 11 per cent chromium. The scale did not show flaking. All of the other specimens showed various degrees of semiadherence. That is, part or all of the outer scale had been rejected in cooling. Alloy C-6 shows part of the outer scale retained, and alloy C-12 shows no outer scale.

Flaking of Oxide Scale:

In determining the corrosion of alloys at high temperatures, the appearance and adherence of the oxide coatings were studied. Because of their observed properties, they were placed into three general categories: (1) adherent, (2) semiadherent, and (3) free-flaking.

As implied by the term itself, adherent scale cannot be removed mechanically, as by prying. Free-flaking scales are those which flake vigorously in small particles and may be thrown a distance of several feet. This phenomenon is attributed to the difference in thermal contraction properties between the metal and its oxide. The other term, semiadherent, falls between the above two.

In a study made at 1800 F., alloys of 11 and 16 per cent chromium containing less than 30 per cent nickel were generally observed to be of the free-flaking type, with the exception of the very low-nickel alloys which appeared to be of the semiadherent and adherent types. Alloys having scales of the free-flaking type may lose 50 per cent or more of their scale by vigorous flaking and retain the remainder as an adherent coating. Most alloys, however, form scales that are combinations of these three general types, and their characteristics may change somewhat with the testing temperature, type of atmosphere, and duration of exposure.

Most of the alloys shown in Fig. 9 are classed as forming semiadherent scales, and most of those in Fig. 10 are classed as free-scaling. Alloy C-2 in Fig. 10 perhaps should be classed as forming an adherent scale.

1000-HR. AIR-CORROSION TESTS AT 1800 F.

The data in this paper so far have been based on the results of tests run for 100 hr. of continuous heating. It was desirable to determine the effect of longer periods of time and to make a comparison between continuous heating and interrupted heating. A third variable has been introduced in which the specimens, in addition to being subjected to interrupted heating, have been subjected to light tumbling which removed some of the scale. These tests have been continued for 1000 hr. and are described briefly as follows:

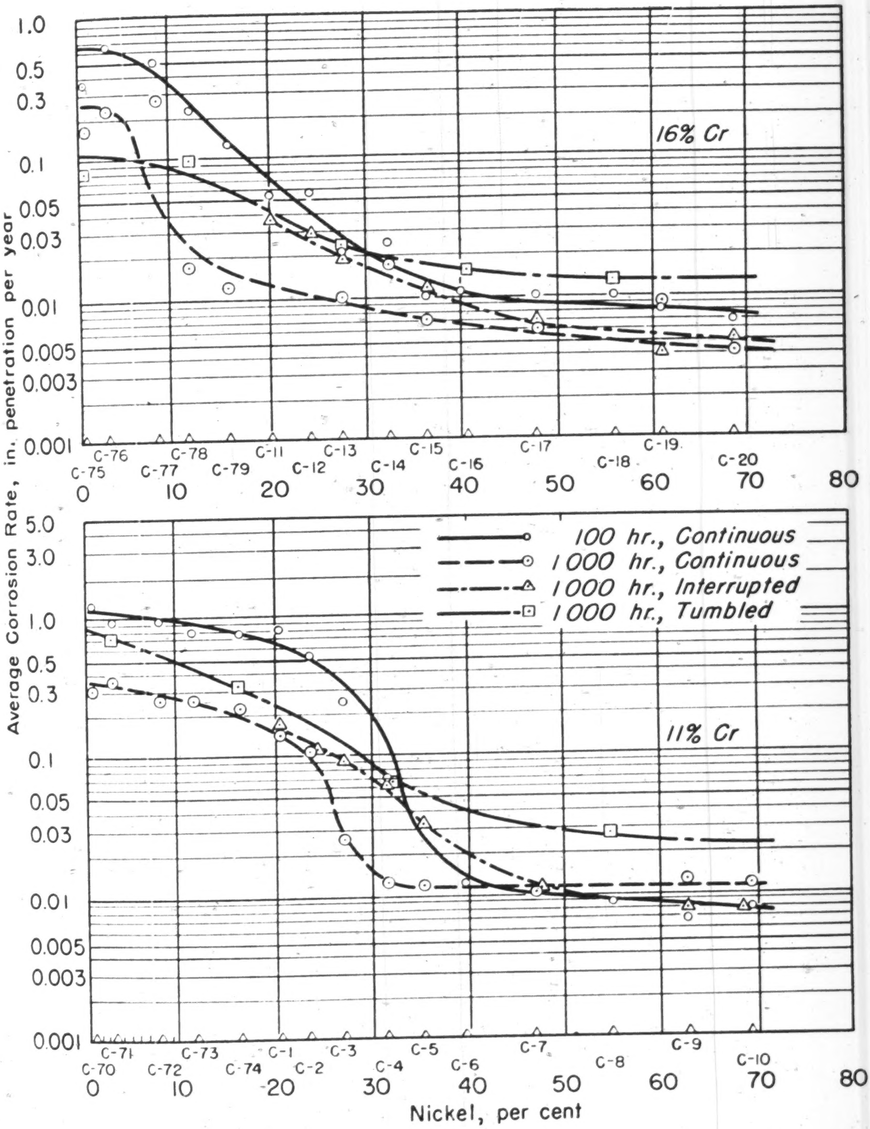

Fig. 12.—Comparison of Corrosion Penetration Rates for 100-hr. and 1000-hr. Tests at 1800 F. Chromium at 11 and 16 per cent with variable nickel contents.

TABLE V.—SUMMARY OF METAL LOSS DATA OF AIR-CORROSION TESTS MADE AT 1800 F. UNDER DIFFERENT EXPOSURE CONDITIONS.

Alloy	Compositions of Alloys, per cent						Calculated Corrosion Rates, in. per year			
								1000-hr. Tests		
	Carbon	Nickel	Chromium	Silicon	Manganese	Nitrogen	100-hr. Tests	Continuous	Interrupted	Interrupted and Tumbled
				11 per cent Chromium Alloys						
No. C-70	0.45	0.13	11.0	1.31	0.78	0.05	1.27	0.306	....	....
No. C-71	0.45	3.0	10.9	1.30	0.76	0.05	0.928	0.371	....	0.740
No. C-72	0.45	8.3	11.0	1.28	0.75	0.06	0.963	0.273	....	....
No. C-73	0.45	12.1	11.0	1.22	0.78	0.05	0.804	0.270	....	....
No. C-74	0.40	16.1	11.0	1.20	0.72	0.05	0.800	0.231	....	0.331
No. C-1	0.34	20.13	11.21	1.21	0.76	0.046	0.855	0.164	0.177	....
No. C-2	0.34	23.66	11.01	1.21	0.76	0.046	0.566	0.110	0.118	....
No. C-3	0.33	27.37	10.84	1.21	0.76	0.046	0.251	0.027	0.091	....
No. C-4	0.35	31.84	11.12	1.28	0.82	0.035	0.073	0.013	0.062	0.066
No. C-5	0.35	35.57	11.06	1.28	0.82	0.035	....	0.013	0.032	....
No. C-6	0.39	39.31	10.77	1.28	0.82	0.035	0.013	....	....	....
No. C-7	0.31	47.34	11.09	1.28	0.84	0.022	....	0.010	0.011	....
No. C-8	0.37	55.09	11.21	1.28	0.84	0.022	0.009	....	....	0.029
No. C-9	0.32	63.03	10.98	1.36	0.90	0.008	0.007	0.014	0.008	....
No. C-10	0.37	69.07	10.87	1.36	0.90	0.008	0.008	0.013	0.008	....
				16 per cent Chromium Alloys						
No. C-75	0.47	0.11	16.0	1.26	0.78	0.03	0.347	0.174	....	0.081
No. C-76	0.43	3.0	16.1	1.23	0.75	0.04	0.661	0.228	....	....
No. C-77	0.45	8.2	15.7	1.20	0.75	0.06	0.499	0.282	....	....
No. C-78	0.46	12.0	15.8	1.22	0.77	0.06	0.222	0.018	....	0.097
No. C-79	0.44	16.2	15.8	1.18	0.69	0.06	0.139	0.013	....	....
No. C-11	0.47	20.00	16.17	1.16	0.80	0.050	0.054	....	0.037	....
No. C-12	0.48	24.10	16.02	1.16	0.80	0.050	0.055	....	0.029	....
No. C-13	0.50	27.55	16.25	1.16	0.80	0.050	0.021	0.010	0.019	0.023
No. C-14	0.45	32.20	15.98	1.24	0.80	0.075	0.024	0.018	....	....
No. C-15	0.45	36.43	15.98	1.24	0.80	0.075	0.010	0.007	0.012	....
No. C-16	0.46	40.34	15.98	1.24	0.80	0.075	0.011	....	....	0.016
No. C-17	0.40	47.95	15.73	1.25	0.84	0.057	0.010	0.006	0.007	....
No. C-18	0.40	56.07	15.86	1.25	0.84	0.057	0.010	....	....	0.013
No. C-19	0.39	60.95	15.73	1.24	0.84	0.032	0.008	0.009	0.004	....
No. C-20	0.42	68.13	15.90	1.24	0.84	0.032	0.007	0.004	0.005	....
				21 per cent Chromium Alloys						
No. C-80	0.44	0.14	20.8	1.31	0.78	0.05	0.015	0.008	....	0.012
No. C-81	0.43	3.0	21.0	1.28	0.73	0.03	0.082	0.007	....	....
No. C-82	0.45	8.2	21.1	1.25	0.75	0.07	0.073	0.008	....	0.039
No. C-83	0.47	12.1	20.8	1.23	0.76	0.06	0.040	0.008	....	....
No. C-84	0.44	16.3	20.7	1.18	0.73	0.06	0.014	0.007	....	0.014
No. C-31	0.46	19.95	20.96	0.76	0.73	0.058	0.015	....	0.016	....
No. C-32	0.46	24.08	20.92	0.76	0.73	0.058	0.015	....	....	....
No. C-33	0.47	28.34	20.85	0.76	0.73	0.058	0.015	0.007	0.006	0.008
No. C-34	0.47	32.32	20.92	1.21	0.77	0.072	0.011	....	....	....
No. C-35	0.47	36.02	21.03	1.31	0.77	0.064	0.010	0.006	0.005	....
No. C-36	0.46	40.08	20.98	1.29	0.76	0.064	0.009	....	....	0.008
No. C-37	0.46	47.90	21.00	1.27	0.77	0.080	0.009	0.004	0.004	....
No. C-38	0.45	55.92	20.77	1.35	0.81	0.067	0.010	....	....	0.008
No. C-39	0.45	61.84	21.22	1.17	0.78	0.054	0.009	0.004	0.004	....
				26 per cent Chromium Alloys						
No. C-85	0.45	0.16	25.8	1.34	0.78	0.04	0.057	0.007	....	0.006
No. C-86	0.43	3.0	25.9	1.32	0.73	0.03	0.073	0.005	....	....
No. C-87	0.45	8.2	25.8	1.30	0.75	0.07	0.024	0.005	....	0.010
No. C-88	0.47	12.1	25.6	1.23	0.77	0.05	0.017	0.005	....	....
No. C-89	0.45	16.0	25.8	1.15	0.74	0.06	0.010	0.004	....	0.008
No. C-41	0.46	20.12	25.47	1.12	0.68	0.046	0.016	0.003	0.007	....
No. C-42	0.47	23.48	26.00	1.23	0.73	0.126	0.011	....	....	0.007
No. C-43	0.47	27.40	25.78	1.22	0.75	0.139	0.012	0.005	0.004	....
No. C-44	0.45	31.80	25.64	1.22	0.72	0.074	0.014	....	....	0.007
No. C-45	0.49	35.94	25.92	1.22	0.74	0.093	0.013	0.005	0.005	....
No. C-46	0.45	40.15	26.08	1.24	0.74	0.087	0.011	....	....	0.007
No. C-47	0.49	48.04	25.93	1.17	0.77	0.083	0.013	0.005	0.005	....
No. C-48	0.42	55.82	25.94	1.27	0.73	0.079	0.008	0.004	0.004	0.006

TABLE V—*Continued*

Alloy	Compositions of Alloys, per cent						Calculated Corrosion Rates, in. per year			
							100-hr. Tests	1000-hr. Tests		
	Carbon	Nickel	Chromium	Silicon	Manganese	Nitrogen		Continuous	Interrupted	Interrupted and Tumbled
31 PER CENT CHROMIUM ALLOYS										
No. C-90	0.47	0.20	30.9	1.30	0.77	0.05	0.052	0.006	....	....
No. C-91	0.44	3.0	30.9	1.22	0.72	0.04	0.035	0.005	....	0.007
No. C-92	0.43	8.2	31.0	1.28	0.77	0.07	0.012	0.003	....	....
No. C-93	0.44	12.1	30.8	1.24	0.77	0.06	0.011	0.003	....	0.006
No. C-94	0.46	15.9	30.8	1.19	0.73	0.06	0.010	0.003	....	....
No. C-51	0.43	20.12	30.71	1.12	0.73	0.062	0.026	0.005	0.005	....
No. C-52	0.44	23.81	30.98	1.13	0.72	0.058	0.035	....	....	0.006
No. C-53	0.43	27.44	31.09	1.17	0.73	0.057	0.028	0.005	0.005	....
No. C-54	0.44	31.28	30.79	1.19	0.73	0.083	0.027	....	....	0.006
No. C-55	0.43	36.42	30.35	1.10	0.71	0.081	0.022	0.006	0.005	....
No. C-56	0.48	40.25	30.68	1.18	0.70	0.114	0.022	....	....	0.007
No. C-57	0.44	48.35	30.62	1.25	0.67	0.093	0.023	0.004	0.006	....
36 PER CENT CHROMIUM ALLOYS										
No. C-64	0.45	31.85	35.50	1.15	0.68	0.079	0.031	0.008	0.004	0.006
No. C-65	0.45	36.18	35.40	1.26	0.69	0.114	0.023	0.005	0.006	....
No. C-66	0.45	39.80	35.68	1.29	0.70	0.091	0.027	0.005	0.005	....

(*a*) The specimens were heated continuously at 1800 F. for 1000 hr. Other conditions for making the tests were the same as those described earlier for the tests run for the period of 100 hr.

(*b*) The specimens were heated as in (*a*) above except that at 24-hr. intervals they were removed from the furnace, cooled to room temperature and returned to the furnace. This cycle was continued for a total of 1000 hr. in the furnace.

(*c*) In this run, the procedure was as in (*b*), except that the specimens were subjected to a tumbling operation for 5 min. to remove loosely adhering scale and weighed before they were reinserted into the furnace.

The compositions of the alloys included in these tests are given in Table V, and it will be noted that alloys at five chromium levels with nickel as a variable have been investigated. The tabulated data are in terms of corrosion penetration calculated to inches per year. Data for 100-hr. tests are included for comparison purposes. The results of these tests are shown graphically in Figs. 12 and 13. Figure 12 relates to alloys at the chromium levels of 11 and 16 per cent with variable nickel contents, while Fig. 13 relates to alloys of chromium contents of 21, 26, and 31 per cent, with variable nickel contents.

11 per cent Chromium.—Referring to Fig. 12 and to alloys containing 11 per cent chromium, comparisons may be made of the rates of penetration under the four testing conditions and as the nickel content varies from zero to 70 per cent. For nickel contents of zero to over 30 per cent, it is evident that the indicated rate of corrosion penetration from the 100-hr. tests is substantially greater than that for the 1000-hr. continuous exposure test. However, at 40 per cent nickel and above, there is little difference in the results as a function of the time of testing. The most significant difference between the results of continuous and interrupted heating is found for alloys of this chromium content when the nickel is between about 20 and about 50 per cent. It is most pronounced between 30 and 40 per cent, with the interrupted exposure showing the higher rate of corrosion penetration. This is attributed to the tendency of alloys in this group to be free scaling so that the

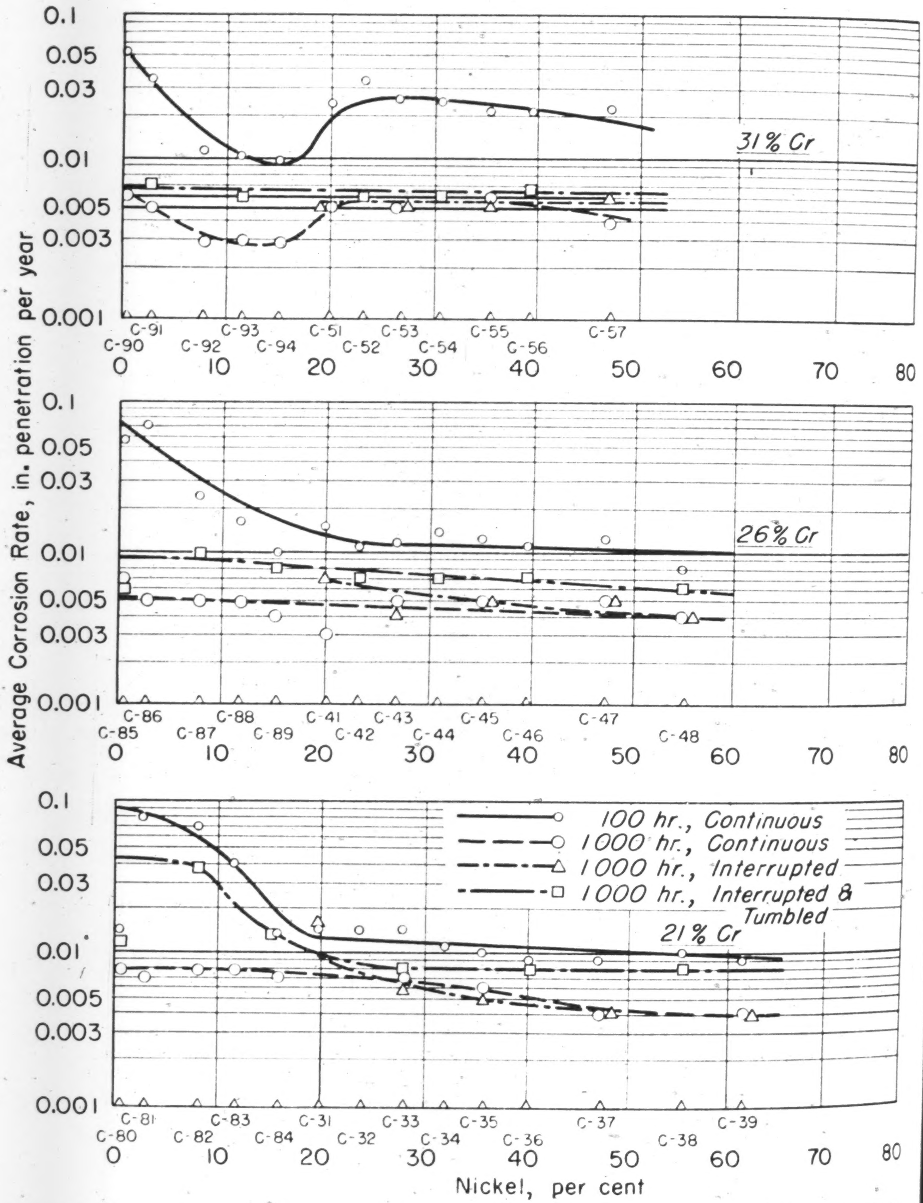

FIG 13.—Comparison of Corrosion Penetration Rates for 100-hr. and 1000-hr. Tests at 1800 F. Chromium at 21, 26, and 31 per cent with variable nickel contents.

parent metal is not fully protected by all the scale formed. Light tumbling showed higher rates of penetration at all nickel contents than either the continuous or interrupted exposure, and for the higher nickel contents gave higher values than those indicated for the 100-hr. tests.

16 per cent Chromium.—Figure 12 also shows the results of tests on alloys of 16 per cent chromium with variable nickel contents. The results of 100-hr. tests indicate substantially higher rates of penetration than the 1000-hr. tests, especially for alloys containing up to 30 to 40 per cent nickel, above which the difference is comparatively small. Data are incomplete for comparisons between continuous and interrupted heating in the 1000-hr. tests, but interrupted heatings show a substantially greater rate of penetration in the range of 20 to 30 per cent nickel, but at the higher nickel contents there were comparatively little differences. The commercial HT alloy (36 per cent nickel, 16 per cent chromium) may be mentioned in connection with this group. For alloys of this composition, it is indicated that there is comparatively little difference between continuous and interrupted heating, and the results of 100-hr. tests may be considered as fairly indicative of longer exposure tests, at least up to 1000 hr. Again, light tumbling in general increased the rate of corrosion penetration.

21 per cent Chromium.—Figure 13 shows the rate of corrosion penetration under the four conditions of testing for 21, 26, and 31 per cent chromium alloys with variable nickel contents.

Considering the 21 per cent chromium alloys, there is a very marked difference in the rates of corrosion penetration as calculated from the 100-hr. test as compared with those from the 1000-hr. test in the nickel-free alloys, with this difference decreasing rather rapidly to about 20 per cent nickel. Above 20 per cent nickel, the rates of penetration for these two conditions of test are parallel, but with the calculated values for the shorter time of testing being somewhat greater. Only a limited comparison can be made between continuous and intermittent heating, but the data suggest that intermittent heating may be somewhat more severe at 20 per nickel and less but there is essentially no difference for the higher nickel contents. Intermittent heating and tumbling give a closely similar value for a nickel-free alloy, but there is a substantial difference with an 8 per cent nickel alloy, with this difference decreasing as the nickel content increases up to about 30 per cent nickel. With 30 per cent nickel and higher, the specimens which were tumbled showed somewhat greater penetration than either the continuous or intermittent heating, but still they gave values somewhat less than those calculated from the 100-hr. test. The tests at different chromium levels are of some interest because alloys such as 20 Cr-25 Ni would be included, and it is indicated that such alloys would show relatively little scaling or only nominal variations in rates of penetration calculated from 100 and 1000-hr. tests.

26 per cent Chromium.—The data for the 26 per cent chromium alloys show quite a marked difference, about 10 to 1, for the corrosion penetration of the nickel-free alloys as calculated from the 100-hr. test as compared with those calculated from the 1000-hr. test. This difference decreases as the nickel content is increased to about 20 per cent nickel, above which the relations are quite consistent. While the data are somewhat limited, it is indicated that the rates of corrosion penetration, as determined by continuous and intermittent heating, are closely similar. Tumbling the specimens increased the rate of corrosion penetration slightly, but the results for the

1000-hr. tests under this condition were in all cases lower than those indicated for the 100-hr. tests. This group of alloys is of interest because it includes the HH type with 25 per cent chromium and 12 per cent nickel. It is to be noted that the results of the 100-hr. tests indicate about four times the penetration indicated by the 1000-hr. tests. This, of course, means that this alloy forms an adherent scale which retards further corrosion penetration.

31 per cent Chromium.—The results of the tests on alloys of 31 per cent chromium with variable nickel contents show that the 100-hr. tests indicate a much higher rate of penetration for all nickle contents than do the 1000-hr. tests. It is to be noted that both of these methods of testing show decreasing rates of corrosion penetration as the nickel content is increased to about 10 per cent and then show an increase in corrosion penetration starting at about 20 per cent nickel. The indications are that continuous and interrupted exposure tests give essentially the same results. For the most part, tumbling had little effect on corrosion penetration, except that the 12 per cent nickel alloy showed greater corrosion penetration than on continuous heating.

Tests of 1000-hr. duration have not been made at 1600 F. or at the higher temperatures of 2000 or 2200 F. It is not considered possible to predict from the tests at 1800 F. what the results would be at either higher or lower temperatures except in some very general way. However, the principle is rather well established that those alloys which form protective scale retard further corrosion penetration, and the rates as calculated from 1000-hr. tests are substantially lower than those calculated from 100-hr. tests, and it seems probable that, in long-periods-of-service tests, such alloys will show up favorably. On the other hand, certain alloys form less protective coatings, and the results of short-time tests are rather closely similar to the results of longer time tests.

SUMMARY

The resistance of iron-nickel-chromium alloys to corrosion in air has been studied at temperatures of 1600, 1800, 2000 and 2200 F. Some iron-chromium alloys have been studied for comparison purposes. Most of the tests were run for 100 hr. of continuous heating in an atmosphere saturated with water vapor at 90 F. Longer tests of 1000-hr. duration have been made on selected alloys under three conditions of test: (*a*) continuous heating, (*b*) interrupted heating, and (*c*) interrupted heating with light tumbling before the specimens were replaced in the furnace.

In addition to showing the data in tabulated form, curves have been drawn to show the effect of nickel and chromium variations, and isocorrosion lines have been drawn on ternary diagrams. At each temperature, the isocorrosion lines show that there is a plateau or region of best corrosion resistance and that this region shifts somewhat as the testing conditions are varied. Three commercial alloys were included in the tests, and their relative corrosion resistance to the other alloys of the series is indicated on the ternary diagrams for the different testing temperatures.

Tests run over a period of 1000 hr. show that, in general, the trends indicated by 100-hr. tests were also found for the longer testing periods, although substantial differences were found in certain alloy compositions with a tendency for the indicated rate of corrosion to decrease as the testing time was increased. These relations are brought out in graphic form and are also shown in the tabulated data. Certain alloy compositions show more rapid corrosion when subjected to intermittent heating and cooling than when heated continuously. The greater rates of corrosion under conditions of intermittent heating are attributed to loss of scale during the heating and cooling operations, especially the latter.

No claim is made that the results of these tests made in air can be used as a basis for the selection of alloys to be used in the more complicated atmospheres encountered in industry. However, it is concluded that the tests made in air for a period of 100 hr. give safe indications of the resistance of these alloys to air corrosion, but with certain alloy compositions, longer exposures give a more positive indication of the service performance.

Acknowledgments:

This paper is based on a research conducted for the Alloy Casting Inst. at Battelle Memorial Inst. Most of the cast-alloy materials in this investigation were prepared by the Research Laboratory of the American Brake Shoe Co., but a few compositions were prepared in the Battelle Memorial Inst. foundry. A brief preliminary description of this work appeared in Alloy Casting Bulletin No. 4, March, 1945. The tests at 2200 F. and the series of alloys below 20 per cent nickel were not included in that report. The authors are indebted to the Alloy Casting Inst. for permission to publish this paper.

DISCUSSION

Mr. Leo Schapiro.[1]—I should like to ask whether the descaling was by the sodium hydride method. If not, would the authors give us a description of the method used.

Mr. C. T. Evans.[2]—This is an excellent piece of work, as far as it goes, but due to the large number of failures which have occurred in heat-resistant alloys over the past twenty years, and which have been unpredictable from weight-loss data, I do not believe the conclusions presented here are justified. I am referring particularly to the type of failure which results from intergranular oxidation. Also, there is graphitization to be considered. Sulfidation likewise cannot be determined from weight-loss studies. I am sure that Mr. Harder is aware of this, and I should like to know what is being done to examine these samples to determine what damage may have occurred from these causes.

Mr. Howard S. Avery.[3]—The difference in behavior under service conditions in comparison with laboratory tests may be important, even if the gaseous environment is the same. Stress is frequently a factor that is absent in laboratory tests.

After creep tests, which are made in air with constant temperature and applied tensile stress, we have noted frequent evidence of intergranular cracking, or incipient failure that is indicated by oxidized grain boundary surfaces on the fracture face after breaking the creep-tested specimen in tension at room temperature. Microscopic examination usually reveals cracks that follow grain boundaries in these cases. They appear to be the first stage of failure in stress-rupture tests. Examples are presented in the accompanying Fig. 14.

There may also appear a decarburized layer extending for a few thousandths of an inch below the surface. There is reason to speculate whether this may contribute to failure, as most of the heat-resistant alloys depend considerably on the strengthening effect of carbon. Whether the weakness of a carbon depleted zone and intergranular oxidation operate singly or together, it is obvious that gaseous corrosion may influence service life by mechanisms superimposed on the cross-sectional area reduction implied by the surface metal loss or penetration values derived from laboratory tests.

TABLE VI.—HOT GAS CORROSION IN AN INDUSTRIAL FURNACE AT 1940 F

Heat No.	Chemical Analysis, per cent						Total Test Period, hr.	Average Corrosion Rate,[a] inches per year
	C	Mn	Si	Ni	Cr	N		
HT-14	0.14	0.72	1.2	34.7	16.4	0.05	8100	0.135
HT-35	0.35	0.67	1.3	34.9	16.0	0.05	8100	0.235
HT-44	0.44	0.74	1.2	34.9	16.4	0.05	8100	0.210
HT-47	0.47	0.82	1.2	34.9	16.0	0.04	5150	0.125
HT-56	0.56	0.80	1.3	34.8	15.9	0.04	5150	0.163
HT-61	0.61	0.76	1.3	34.6	16.1	0.05	5150	0.173
HT-70	0.70	0.81	1.3	34.7	16.5	0.05	5150	0.155
HH-32	0.32	0.54	1.2	12.8	26.7	0.16	8100	0.155
HH-31	0.31	0.55	1.2	11.2	26.8	0.16	8100	0.095
C-4	0.35	0.82	1.28	31.8	11.1	0.04	2110	0.390
C-14	0.45	0.80	1.24	32.2	16.0	0.08	2110	0.120
C-16	0.46	0.80	1.24	40.3	16.0	0.08	2110	0.070
C-34	0.47	0.77	1.21	32.3	20.9	0.07	2110	0.040

[a] For 50 per cent operation with gas and 50 per cent with fuel oil.

The gaseous environment may also be an important variable. The accompanying Table VI includes some data from a field test made under industrial service conditions. The alloys, unstressed, were exposed in a furnace operating intermittently at 1940 F. The specimens, some of which are identical in composition with alloys included in the Alloy Casting Institute program, were in the form of $\frac{1}{2}$ by 1 by 2-in. blocks welded at one end to the bottom of a heat-resistant alloy tray whose sides were high enough to protect against

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[1] Chief Metallurgist, Douglas Research Labs., Douglas Aircraft Co., Inc., Santa Monica, Calif.

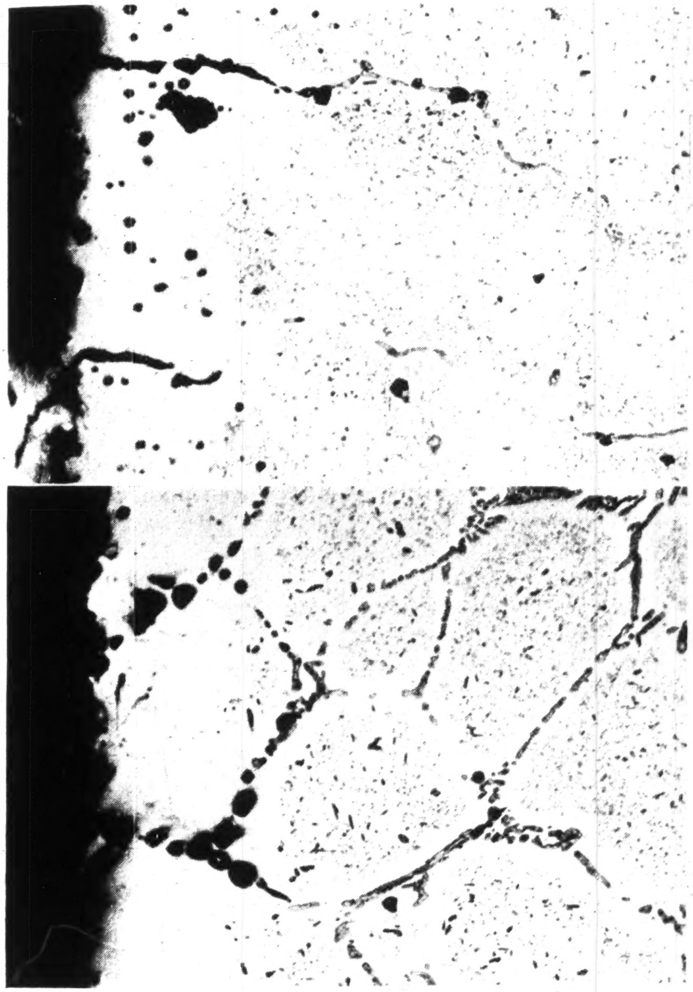
[2] Chief Metallurgist, Elliott Co., Jeanette, Pa.

[3] Research Metallurgist, American Brake Shoe Co., Mahwah, N. J.

Chemical Analysis, per-cent

C	Mn	Si	Ni	Cr	N
0.29	0.72	1.28	34.4	16.2	0.044

Creep tested 630 hr. at 1800 F. and 2000 psi. Note the intergranular attack and the surface decarburization.



Chemical Analysis, per-cent

C	Mn	Si	Ni	Cr	N
0.56	0.71	1.22	34.7	16.4	0.036

Creep tested 1003 hr. at 1800 F. and 2000 psi. Note the different type of surface attack and the similar decarburization.

FIG. 14.—Grain Boundary Surface Attack During Creep Testing of HT Alloys in Air (× 250)
Etchant: Hot Alkaline K_4FeCy_6

direct flame impingement. The tray was rotated at intervals, during shutdown periods, to equalize exposure of the various specimens. The furnace was fired chiefly with natural gas, which has a low sulfur content, but at intervals a fuel oil (Standard Oil Co. of Indiana Industrial Fuel Oil Nos. 350 or 125) was substituted.

A comparison of the periods of gas firing with those when oil was used indicates more severe corrosion with the latter. For 100 per cent gas firing, the rate for the HT alloys is about 0.08 in. per year surface metal loss, while with oil fuel 50 per cent of the time the rate is 0.18 ± 0.06 in. per year.

The influence of chromium and nickel is shown clearly by alloys C-4, C-14, C-16, and C-34, confirming the conclusions of Messrs. Brasunas, Gow, and Harder. The corrosion rates at 1940 F. fall between those at 1800 and 2000 F. in air. Precise agreement is not expected because of the difference in furnace atmospheres.

If the authors would indicate the reproducibility of their test results and the probable error of the reported data it would be very helpful in the interpretation of hot gas corrosion results. In their Fig. 1, alloy C-14 at 2000 F. is plotted at a penetration rate of 0.2 in. per year while the correlation line is drawn through about 0.09 in. per year. Alloy C-12 deviates by a comparable amount in the opposite direction. This suggests that ± 120 per cent deviation from the average may be encountered.

MESSRS. BRASUNAS, GOW, AND HARDER (*authors' closure by letter*).—The authors are pleased to have received discussions of their paper from Messrs. Leo Schapiro, C. T. Evans, and Howard S. Avery. Mr. Avery's contribution regarding the degree of severity of corrosion of a flue-gas atmosphere upon certain alloys similar in composition to those used in our air-corrosion tests makes a valuable addition to our paper.

In reply to Mr. Schapiro, the sodium hydride method was not used. A method described by Mr. Heron in the S.A.E. *Journal* (see footnote 2 of the text) was found to be very satisfactory. In brief, a molten salt bath, consisting of 60 per cent sodium hydroxide and 40 per cent sodium carbonate, was heated to 1000 F. in a steel container which was connected to the positive pole of a 6-v. storage battery and served as an anode. The specimen, connected to the negative pole of the battery, became the cathode when submerged in the salt bath. A current density of about 400 amp. per square foot of specimen surface area was generally maintained for approximately 2 min., but the time interval was varied in certain instances, depending upon the adherence and extent of scale formation. The specimen was then quenched directly into water.

Scale removal results partly from the thermal shock of the descaling operation, but mainly from the electrolytic action.

In regard to Mr. Evans' remark to the effect that weight-loss data have not generally predicted failures that have occurred in heat-resistant alloys in service applications, we most fully agree. We also are of the opinion that certain alloy compositions which are prone to intergranular or interdendritic oxidation may be damaged more severely by this type of attack than by general surface scaling. However, a generalization on this point covering the whole realm of Fe-Ni-Cr alloys cannot be made.

We have observed relatively shallow corrosion penetration along the primary carbides of the structure for certain alloy compositions exposed in the elevated-temperature air-atmosphere corrosion tests without stress being applied to the material.

Figure 15 shows the regions of alloy composition in which various forms of subsurface corrosion attack have been observed by microscopic study of transverse sections of the specimens from the 1800 F. air-oxidation tests. Figures 16 and 17 illustrate different types and degrees of subsurface corrosion attack that have been observed. Undoubtedly all such forms of subsurface corrusion attack have some damaging influence on the strength property of the metal in the layer in which they occur. Oxide penetration might well be considered as providing a notch effect and thereby serving to accelerate failure under tensile stresses, shock, or alternate thermal stresses. Decarburization would be expected to lower the resistance of the alloy to flow (creep) under stress since the alloys are known to have maximum creep resistance at an optimum carbon content. However, it could well be that having a layer of metal of improved plasticity on the surface as a result of decarburization may be of benefit rather than a detriment under certain conditions of stressing.

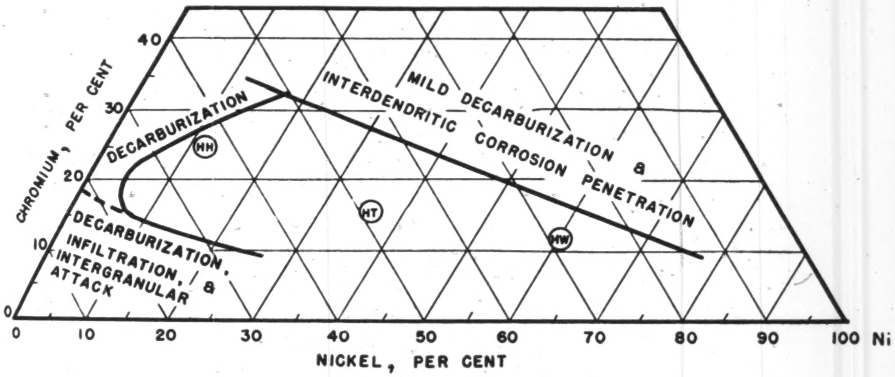


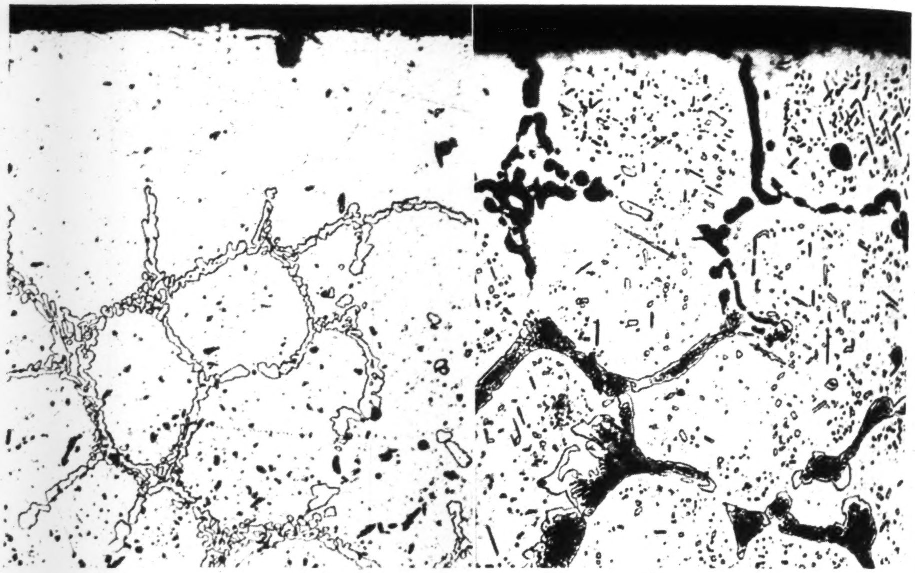
Fig. 15.—Types of Subsurface Corrosion Attack Found in Various Alloy Regions in Air Atmosphere at 1800 F. for 100 hr.

The region of alloy compositions in which substantially no subsurface corrosion attack was evident includes the three commercially important alloy types, HH (25 per cent chromium—12 per cent nickel), HT (35 per cent nickel—16 per cent chromium), and HW (60 per cent nickel—12 per cent chromium). In general, as the composition departs from the region of substantially no subsurface corrosion attack the intensity of attack was found to increase.

It is to be recognized that, while stress, and especially alternating stress when applied to a structural member, is undoubtedly the primary cause for practically all service failures of heat-resistant alloy structural members, corrosion in the form of general surface scaling and subsurface corrosion attack also are contributing causes through acting to reduce the cross-section of the member and thereby increasing the unit stress or providing points of stress concentration. There are also other features of the microstructure of the alloys to be considered, such as the distribution, continuity, and stability of the carbide phase, the physical and mechanical properties of this phase in relation to that of the matrix solid solution, and also the cohesion or bond between the carbide and the matrix alloy.

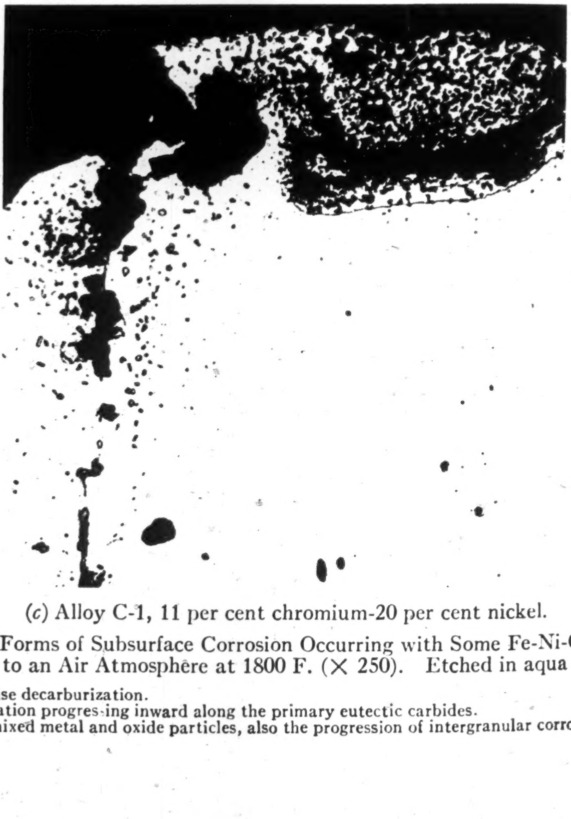
In regard to the statement that graphitization should considered, we wish to state that we have not observed it to occur in the alloy compositions reported upon at this time.

The present paper has been intentionally limited to only one mode of



(*a*) Alloy C-85, 26 per cent chromium-0 per cent nickel.

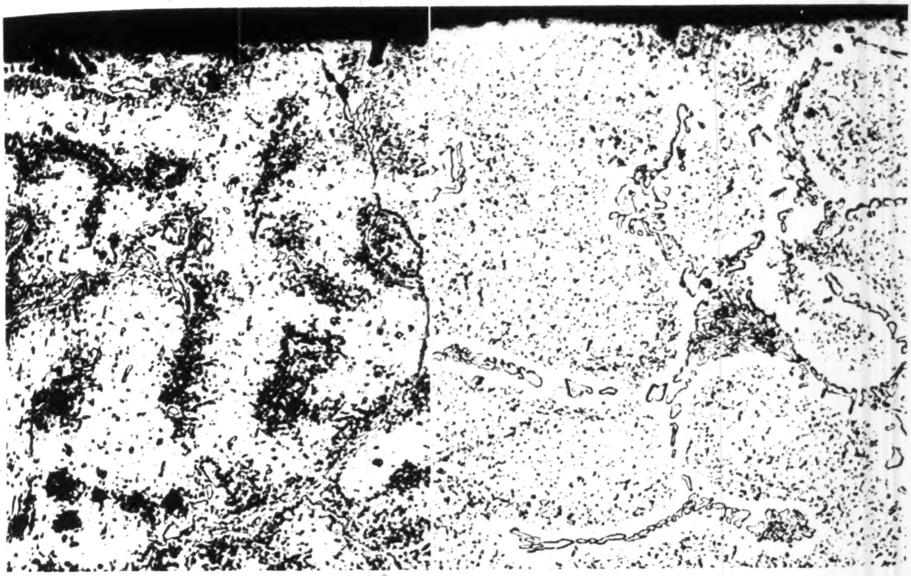
(*b*) Alloy C-55, 31 per cent chromium-36 per cent nickel.



(*c*) Alloy C-1, 11 per cent chromium-20 per cent nickel.

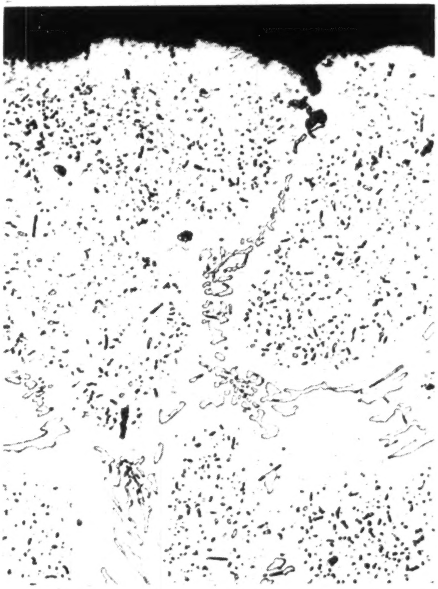
FIG. 16.—Various Forms of Subsurface Corrosion Occurring with Some Fe-Ni-Cr Alloys upon Exposure to an Air Atmosphere at 1800 F. (× 250). Etched in aqua regia.

(*a*) Note zone of intense decarburization.
(*b*) Note oxide penetration progressing inward along the primary eutectic carbides.
(*c*) Note the area of mixed metal and oxide particles, also the progression of intergranular corrosion.



(*a*) Alloy C-26, regular HH alloy, 25 per cent chromium—12 per cent nickel.

(*b*) Alloy C-22, regular HTalloy, 16 per cent chromium—36 per cent nickel.



(*c*) Alloy C-10, closely resembling an HW type, 11 per cent chromium—68 per cent nickel.

FIG. 17.—Subsurface Corrosion of Some Commercial Fe-Ni-Cr Alloys Exposed to an Air Atmosphere at 1800 F. for 100 hr. Etched in aqua regia (× 250).

Note very slight extent of decarburization and interdendritic corrosion on these commercial type alloys.

damage encountered in high-temperature application of the alloys, that of oxidation scaling. It is intended to serve mainly to show the realm of Fe-Ni-Cr alloy compositions of optimum oxidation scaling resistance as a function of exposure temperature. It is clearly revealed by the isocorrosion lines on the ternary diagrams of Figs. 4, 5, 6, and 7 that failure by scaling alone can occur with improper selection of alloy composition, and that the realm of alloys of optimum corrosion resistance decreases as the maximum temperature requirement increases.

We wish to caution against using the information presented on air oxidation when considering the application of alloys for use in contact with other ambient atmospheres. The data submitted by Mr. Avery, however, regarding corrosion in a flue gas provided by burning natural gas present a similar picture of the relation of alloy composition to scaling resistance to that of our work. Such an atmosphere, which would consist mainly of CO_2, water vapor, and nitrogen, would be expected to have an oxidizing scaling action similar to that of air.

In regard to the question of reproducibility of data raised by Mr. Avery, repeat tests have more closely duplicated original tests than implied by the most divergent comparison he has made. For example, in air-corrosion Tests A and B at 2200 F., the following are typical duplicate data:

Alloy	Metal Loss, g. per sq. in. per day		
	Test A	Test B	Average
No. C-81	0.0467	0.0621	0.0544
No. C-82	0.0278	0.0312	0.0295
No. C-83	0.0214	0.0221	0.0218
No. C-84	0.0186	0.0187	0.0187
No. C-34	0.0662	0.0776	0.0719
No. C-36	0.0415	0.0521	0.0468
No. C-37	0.0204	0.0344	0.0274

The deviation of actual data from average data is in the vicinity of 25 per cent. The isocorrosion lines on the ternary diagrams are smooth curves drawn to satisfy the corrosion values obtained for a range of alloy compositions. It is considered likely that deviations such as Mr. Avery has pointed out are the result of a few of the specimens containing microvoids or also variations in the amount of the minor elements such as carbon, silicon, or manganese are entering into the picture.

Mr. Avery has mentioned noting frequent evidence of intergranular cracking by conducting microscopic examination

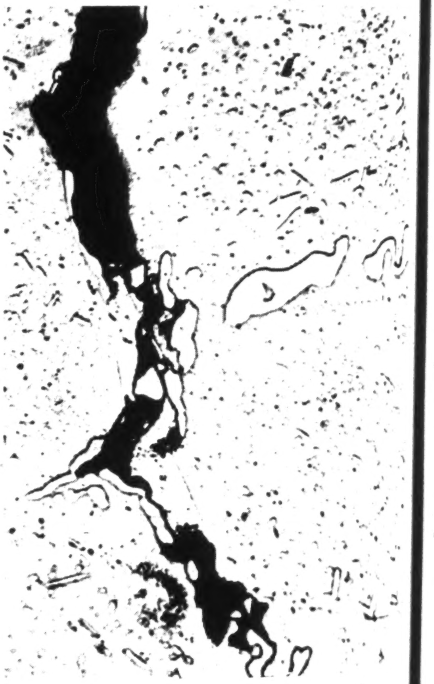


FIG. 18.—Intergranular Cracking Located Adjacent to the Interdendritic Primary Carbides (× 800). Specimen fractured in creep test at 1800 F. Alloy of 0.43 per cent carbon, 14.2 per cent chromium, 34.0 per cent nickel, 1.38 per cent silicon, and 0.90 per cent manganese.

of sections from fractured creep test bars, which is indicated by oxidized grain-boundary surfaces on the fractured faces. We also have observed that the rupturing in such tests follows along the interdendritic network of primary carbides in the structure as shown in Fig. 18. We, however, have never found evidence that deep intergranular corrosion proceeded ahead of the fracturing, but rather that rupture along the carbide network went first and that the newly exposed surface was then oxidized. Fracturing could initiate at a point of stress concentration on the surface of the bar. This could be the result of an unusually shaped carbide or concentration of carbides at a point or over an area of the surface or to interdendritic corrosion penetration being more severe at some point or area as a result of structural nonuniformity. From what we have observed of incipient cracks being present in creep bars, which apparently were not continuations of cracks reaching the surface, it is a matter of speculation as to whether fracturing does in all instances initiate at the metal surface.

INFLUENCE OF STRAIN RATE AND TEMPERATURE ON THE MECHANICAL PROPERTIES OF MONEL METAL AND COPPER*

By D. J. McAdam, Jr.,[1] G. W. Geil,[1] and D. H. Woodard[1]

Synopsis

The third stage of creep may be initiated by the formation of microscopic cracks, predominantly intercrystalline. Cracking occurs when the rising true stress reaches a technical cohesion limit determined by the temperature, strain rate, and amount of plastic deformation. Progressive disintegration thus begins and continues to complete fracture. Both the second-stage flow stress and the cohesion limit increase with decrease in temperature and with increase in the strain rate. Decrease in temperature or increase in strain rate increases the cohesion limit to a greater extent than the flow stress; the ductility thus increases. A study is made of the influence of temperature and the strain rate on the second-stage flow stress, ultimate stress, technical cohesive strength and ductility. By combining these results with previously published data, the view is broadened to include a temperature range from −188 C. to the melting point for monel metal and copper.

In previous papers, it has been shown that the technical cohesion limit and the flow stress are similarly affected by either the stress system, amount of plastic deformation, or temperature. In this paper, it is shown that these two strength indices are similarly affected by the strain rate.

Results of a general investigation at the National Bureau of Standards of factors involved in the fracture of metals have been presented in a number of papers (**11**–**19**).[2] These papers describe studies of the influence of the stress system, temperature, and the amount of plastic deformation on the breaking stress and other mechanical properties. Strain rate, another important factor, has not been considered, except briefly in one of the papers (**17**). The present paper discusses the relation between creep rate, temperature, flow stress, breaking stress, and ductility, and then presents a general view of the influence of strain rate and temperature on the mechanical properties of monel metal and oxygen-free copper between −188 C. and the melting points. The monel metal is the same that was used in the creep tests described in a previous paper (**3**). In that paper, however, attention was confined to the relation between stress, temperature, and a characteristic creep rate, corresponding approximately to the rate in the "second stage" of creep. The relation between the breaking stress and the creep rate was not considered. Both the copper and monel metal have also been used in other phases of the general investigation of technical cohesive strength of metals (**13**–**14**).

Apparatus, Materials, Specimens

A description of the creep test apparatus, extensometer, specimens, methods

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[1] Chief, Section on Thermal Metallurgy, Metallurgist, and Assistant Metallurgist, respectively, National Bureau of Standards, Washington, D. C.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 917.

TABLE I.—DESCRIPTION OF METALS.

Metal	Designation	Mechanical Treatment		Rod Diameter, in.	Chemical Composition, per cent						
		Method	Reduction of Area, per cent		Carbon	Iron	Nickel	Copper	Manganese	Silicon	Sulfur
Oxygen-free copper	N	Cold-rolled	75[a]	0.875	....	....	.....	99.97	....	....	.....
Monel metal	G	Cold-drawn	40[a]	0.875	0.18	1.24	69.02	28.46	0.94	0.10	0.007

[a] Cold-worked without intermediate annealing.

TABLE II.—HEAT TREATMENT IN LABORATORY.

Metal	Designation	Temperature, deg. Fahr.	Time Held, hr.	Cooled in
Oxygen-free copper	N-8	800	5	air
Monel metal	G-14	1400	1	water

of loading, and measurement of extensions is given in a previous paper (3). A description of the metals and their heat treatments is given in Tables I and II, respectively The metals were tested in both cold-worked and annealed conditions. The oxygen-free, high-conductivity copper was supplied by the Scomet Engineering Co. through the cooperation of Sidney Rolle, Assistant Manager. The monel metal was supplied by the International Nickel Co. through the cooperation of the late A. J. Wadhams, Director of Research.

FRACTURE DURING THE THIRD STAGE OF CREEP

A creep test under constant load may be divided into three stages. In the first stage, the creep rate decreases; in the second stage, it is nearly constant; in the third stage, it increases. The duration of the second stage may vary considerably. In many conditions of temperature and creep rate the third stage begins when the rate of work-hardening decreases below a certain value. The evidence now to be presented, however, indicates that the third stage sometimes begins with the formation of microscopic cracks. The discovery of microscopic cracks, predominantly intercrystalline, in some specimens of cold-drawn monel metal that had been extended into the third stage of creep, led to an investigation of the conditions that cause such cracks. As similar cracks were later found in cold-rolled oxygen-free copper, this metal was included in the investigation.[3]

As the cracks appeared early in the third stage of creep, and as this stage was reached after only slight plastic extension, the ductility of these previously cold-worked metals evidently was slight at the beginning of the progressive fracture, whereas the same metals in an ordinary tension test at room temperature failed by abrupt transcrystalline fracture after much plastic deformation (64 per cent reduction of area for the monel metal and 75 per cent for the copper). The evidence thus indicated that the lowered ductility resulted from the combined effects of the higher temperatures and the slower strain rates in the creep tests. The evidence that the ductility tends to decrease with increase in the temperature and with decrease in the strain rate is contrary to the prevalent view. For example, Morkovin proposed to obtain brittle fracture of copper by straining at a very high rate (22). High-speed tests of unnotched specimens at room temperature, however, have given no support to this view, and have indicated that increase of the strain rate generally does not cause decrease of ductility (6, 21). Moreover, decrease of temperature to −188 C. generally does not cause decrease of the ductility of face-centered cubic metals. The behavior of ferritic steels is not characteristic of metals in general.

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[3] There are scattered reports mentioned in the literature of the finding of intercrystalline cracks in metals subjected to prolonged loading, especially at elevated temperatures. No general significance, however, has been attached to such observations (8). In steels, such cracks generally have been attributed to deposition of microconstituents on the grain boundaries.

As the ductility of a metal depends on the difference between the initial flow stress and the breaking stress, the evidence of lowered ductility at slow strain rates indicates that a decrease in the strain rate decreases not only the flow stress and ductility but also the resistance to fracture (technical cohesive strength). In the present investigation, a study was made of the relation between creep rate, temperature and stress at the beginning of fracture. A study was also made of the progress of slow fracture during creep of monel metal and copper.

In a creep test under constant load, the increasing creep rate in the third stage tends to cause an increase of ductility, because as previously explained, the increase in the technical cohesive strength with the strain rate is somewhat greater than the increase in the flow stress. This increase in the creep rate, consequently, tends to retard progressive fracture during the third stage. To study the effect of creep rate and temperature on progressive fracture, therefore, the rate beyond the beginning of the third stage should be held approximately the same as the rate in the second stage. As no apparatus for automatic control of the strain rate was available, an approximately constant rate was maintained by repeatedly decreasing the load. This procedure was found sufficiently accurate for a study of the progress between initial fracture and complete fracture (rupture).

Figure 1 shows typical curves representing the relation between extension and time during creep tests of monel metal. The beginning of the third stage and the point of first weight-removal are indicated in each of the creep-test curves. During the third stage, the creep rate was controlled approximately by repeated weight removals, so that the controlled rate V_c usually was only slightly higher than the minimum rate V_M at the beginning of this stage. After each test, the specimen was photographed, and longitudinal sections were made for microscopical examination. Typical photo-micrographs are shown in Figs. 2, 5, 6, and 7, and photographs of the specimens are shown in Figs. 3, 4, 8 and 9.

All the monel metal specimens showed extensive cracking, which was predominantly intercrystalline (Fig. 2). As shown, the general disintegration of the metal evidently tends to increase with increase in the temperature and with decrease in the strain rate. For each temperature, specimens tested at the highest rates show the least progress of general disintegration, but show a few relatively wide cracks, some of which appear to be transcrystalline. The wide cracks are surrounded by regions of considerable grain distortion. In such specimens, as will be shown in subsequent discussion, the total distortion at complete fracture is greater than in the specimens that show greater general disintegration.

A few of the copper specimens, especially those tested at low temperatures and relatively high strain rates, show no evidence of microscopic cracks. Cracks were found, however, in the specimens tested at slow rates, even when the temperature was as low as 110 F. Typical photomicrographs obtained from specimens with microscopic cracks are shown in Figs. 5, 6, and 7. The grain size of the copper was much larger than that of the monel metal. Comparison of the photomicrographs of the two metals, with allowance for the difference in magnification, shows that the cracks are fewer but generally larger in the copper than in the monel metal. Examination showed that the number of cracks tends to increase with increase in the temperature and with decrease in the strain rate. Most of the cracks in the copper specimens were intercrystalline, but a few were transcrystalline.

The photomicrographs of the cracked specimens of monel metal and copper (Figs. 2, 5, 6, and 7) were studied in correlation with the photographs of the corresponding specimens in Figs. 3, 4, 8, and 9 and with extension-time curves such as those in Fig. 1. Although all the specimens had been extended beyond the beginning of the third stage of creep, many of them did not contract locally even when extended to fracture. More specimens would have contracted locally, however, if the creep rate had been allowed to accelerate. For each temperature used in the creep tests of monel metal, the specimen tested at the highest strain rate showed some local contraction (Figs. 3 and 4). Local contraction disappeared, however, with decrease in the strain rate. The tendency to local contraction was greater with the copper (Figs. 8 and 9) than with monel metal. The greatest local contraction was found in the copper specimen tested at 110 F. with a creep rate of 0.0018 per cent per hour (Fig. 8). Another copper specimen tested at the same temperature at a slower rate, however, was extended well into the third stage (but not to complete fracture) without local contraction (Fig. 8). Of the two copper specimens tested at 375 F. (Fig. 9), only the one tested at the faster rate shows local contraction. For both monel metal and copper, the tendency to local contraction evidently decreases with increase in the temperature and with decrease in the strain rate. The factors governing the instability involved in local contraction will be discussed later in connection with the stress-strain diagrams now to be considered.

In specimens that contract locally before fracture, the number of cracks tends to increase in the notched portion with approach to the surface of complete rupture. This is illustrated in Fig. 7 by a comparison of photographs (*a*) and (*b*). Photograph (*a*) shows a region $\frac{1}{4}$ in. from the surface of rupture. Photograph (*b*) shows a region next to that surface. The two photographs thus show the beginning and ending of the progressive disintegration that occurred during the third stage of creep. These photographs should be compared with the photograph of the specimen (Fig. 8).

FLOW STRESS AND TECHNICAL COHESION LIMIT

Figures 10 to 16 show curves of variation of the flow stress with plastic extension. In a creep test under a constant load, the per cent increase of the "true" stress (the stress based on the current area of cross-section) is equal to the per cent extension, as long as the specimen extends without local contraction. As the monel metal specimens (Figs. 10 to 13) and some of the copper specimens (Figs. 14 to 16) were tested under a constant load throughout the first two stages of creep, the variation of the true stress with extension of these specimens was linear until the first weight removal, as illustrated in the figures. For some of the copper specimens, the load was increased during the first stage of creep, and then kept constant during the second stage. For a few specimens, changes of load were made (through misunderstanding) during the second stage of creep; the stress-strain diagram at the entrance to the third stage, therefore, is not straight. From such a test, only an approximate estimate can be made of the relation between creep rate, temperature, ductility, and the stress at the beginning of fracture.

The transition from the second to the third stage was generally more abrupt for the copper than for the monel metal. In most of the graphs for copper (Figs. 14 to 16), therefore, the point of first weight removal is indistinguishable from the beginning of the third stage.

With the repeated weight removals (to hold the strain rate approximately constant) all but three of the graphs in Figs. 10 to 16 take a general downward course. In Fig. 10 the graphs for monel metal tested at 700 F. and the graph for monel metal tested at 750 F. continue to rise after the first weight removal, but eventually turn downward.

The filled-in triangles in Figs. 10 to 16 indicate technical cohesion limits[4] that initiated the third stage of creep. The disintegration, at a constant strain rate may continue without local contraction until the specimen fails abruptly by complete fracture, as it does in some of the specimens represented in Figs. 10 to 16. In some conditions, however, local contraction may begin some time after the beginning of disintegration. Evidence for this may be found by comparing specimens that have been extended into the third stage, but not to complete fracture, with other specimens that have been extended to complete fracture. For example, a copper specimen that was extended into the third stage at 110 F. and at a minimum creep rate of 0.00028 per cent per hour had begun to disintegrate (Fig. 5) but had not begun to contract locally (Fig. 8). Another specimen, which was extended to complete fracture at a slightly higher temperature (130 F.) and at practically the same creep rate (0.00027 per cent per hour), had begun to disintegrate (Fig. 5) and had contracted locally (Fig. 8). The disintegration in this specimen, therefore, initiated the third stage of creep and the local contraction began later. A similar sequence of cohesion limit and local contraction probably occurred in some of the other specimens that contracted locally. The evidence thus indicates that the third stage of creep may be initiated by progressive disintegration starting when the rising true stress reaches the technical cohesion limit corresponding to the strain rate, temperature, and amount of plastic deformation.

In some conditions, however, the third stage begins without progressive disintegration; the beginning then is not determined by a cohesion limit, but by the work-hardening rate and the creep rate. Plastic deformation may continue without progressive disintegration until the specimen fractures abruptly, as it generally does in an ordinary tension test at room temperature. Disintegration, however, may begin at some time after the beginning of the third stage. Evidence for such a sequence may be found in Fig. 10. Each of the stress-strain curves in this figure continues its general upward course, though with decreasing slope, for some distance beyond the point representing the first weight removal. The beginning of the third stage, therefore, probably was not determined by a technical cohesion limit, but disintegration began later, probably when the stress-strain curve reached its highest point (Fig. 10). All the other stress-strain curves for monel metal (Figs. 11, 12, and 13) take a general downward course immediately after the first weight removal; the evidence thus indicates that the third stage of creep of these specimens was initiated by progressive disintegration such as that shown in Fig. 3.

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[4] By definition, a technical cohesion limit is the technically estimated resistance to fracture, the breaking load divided by the sectional area at the beginning of progressive fracture.

FACTORS DETERMINING THE LIMIT OF STABILITY FOR TENSILE EXTENSION

Consideration will now be given to the factors initiating the third stage of creep in the absence of progressive disintegration, and the factors determining the limit of stability for tensile extension (beginning of local contraction). Local contraction begins when dS/S becomes less numerically than dA/A, when S is the true stress and A is the corresponding area of cross-section. As long as dS/S is greater than dA/A any cross-section that becomes slightly smaller than the adjacent sections becomes stronger and the local contraction stops. When dS/S becomes less than dA/A, however, local contraction begins and continues. In an ordinary tension test at room temperature, the ratio of dS/S to dA/A generally depends almost entirely on the rate of work hardening. In a creep test, however, the local increase in the strain rate due to incipient local contraction causes a local increase in the flow stress, and thus tends to postpone the beginning of continuous local contraction.

Any flow-stress increment, ΔS, during plastic deformation, therefore, may be viewed as the sum of two distinct increments. One of these is the increment $\Delta_1 S$ due to work hardening, the other is the increment $\Delta_2 S$ due to any general or local increase in the strain rate, V. Local contraction begins when: $\frac{d_1S}{S} \Big/ \frac{dA}{A} + \frac{d_2S}{S} \Big/ \frac{dA}{A}$ becomes less than 1.0. These two ratios will be designated m and n, respectively. The value of n is proportional to $\frac{d_2S}{S} \Big/ \frac{dV}{V}$, which will be designated P. The relation between P and n is given by the equation $n = \frac{PdV}{V} \Big/ \frac{dA}{A}$. The variation of P with strain rate and temperature is illustrated by the curves in Figs. 24 and 26, which will be discussed in more detail later. The slope of a curve at any point, with allowance for the difference between the ordinate scale and abscissa scale, is a measure of the corresponding value of P. As shown in these figures, P increases with increase in temperature and with decrease in the strain rate. For incipient local contraction moreover, $\frac{dV}{V} \Big/ \frac{dA}{A}$ would increase with decrease in the strain rate. The ratio n, therefore, increases with increase in the temperature and with decrease in the strain rate.

As illustrated by curve F_0 of Fig. 17(*a*), the increase of the flow stress with the strain under a constant load is linear until local contraction begins. Moreover, the linear variation is such that dS/S is equal numerically to dA/A. The linear variation is the resultant of the curvilinear variation of the flow stress due to work hardening and the opposite curvilinear variation due to the decrease in the strain rate (Fig. 17(*b*)). After the strain rate has become practically constant, the continued linear variation of the flow stress with strain implies that d_1S/S has now become equal to dA/A and that m is 1.0. As long as m remains at this value, $m + n$ is greater than 1.0 and local contraction cannot begin. The linear variation of the flow stress, however, will be terminated when the third stage is reached and weight removals begin for the purpose of holding the strain rate constant. The slope of the stress-strain curve thus decreases as in Fig. 10 or Fig. 17(*a*). Eventually $m + n$ becomes less than

1.0 and local contraction begins. If no weights are removed during the third stage, the continuous increase in the strain rate causes a continuous decrease in P, and eventually $m + n$ becomes less than 1.0.

TRUE STRESS AT COMPLETE FRACTURE

For the monel metal specimens tested at temperatures above 700 F. and at the slower strain rates (Figs. 11, 12, 13), the true stress at complete fracture is about the same whether it is calculated on the assumption that the extension is uniform or by dividing the final load by the area of cross-section at complete fracture. For some specimens, the latter value was slightly smaller than the former because in these specimens the actual decrease in sectional area at complete fracture was less than the decrease calculated from the extension. This discrepancy probably is due to increase in the volume of the specimen because of the voids formed during the disintegration. For the monel metal specimens tested at the faster strain rates or at the lower temperatures, the breaking stress value is much greater when calculated from the final load and the cross-section at complete fracture than when calcuated on the assumption that the specimen extended uniformly to fracture. This discrepancy is due to the local contraction, and is considerable even when the local contraction is apparently slight.

For the copper specimens (Figs. 14, 15, and 16), the stress value for complete fracture was aways greater when calculated from the final load and cross sectional area than when calculated from the total extension on the assumption that the specimen did not contract locally. This discrepancy varies with the amount of the local contraction. Even at the highest temperature and slowest strain rate, the local contraction was not reduced to as low values for copper as for monel metal, probably because the disintegration by internal cracking was less for the copper than for the monel metal. The less extensive disintegration of the copper may be attributed partly to the much larger grain size of the copper, and partly to the fact that cracks spread much less readily in the softer metal. Even in an ordinary tension test of oxygen-free copper, the true breaking stress cannot be determined accurately because the crack after starting near the axis does not spread rapidly to the circumference. Consequently the metal continues to extend at the rim of the section, thus giving fictitiously high breaking stress values (13). An illustration of this behavior may be found in Fig. 15, where a calculated stress for complete fracture is given as 92,000 psi., although the correct value for the breaking stress at room temperature is about 80,000.

Local contraction is accentuated by the increasing strain rate during the contraction. The actual strain rate increases continuously even though the nominal rate remains constant. The extension rate, moreover, sometimes increased considerably during the last stage of creep, because the load could not generally be reduced fast enough to hold the extension rate constant. The plastic deformation and stress at complete fracture thus are greater than they would be if the strain rate had been held at the desired constant value. Therefore, when the specimen has contracted locally in a creep test, the indicated relatively high stresses at complete fracture (R values in Figs. 10 to 16) have little significance. When the specimen has not contracted locally, the decrease of the true stress due to repeated weight removals during the third stage of creep is a measure of the disintegration by internal cracking. The most important breaking stress value obtained in a creep test, however, is the stress at the beginning of progressive disintegration (the technical cohesion limit).[4]

Effect of the Strain Rate and Temperature on Ductility, and Their Differential Effect on Flow Stress and Technical Cohesion Limit

Values of ductility derived from Figs. 10 to 16 have been used in a study of the relation between the strain rate, temperature, and initial breaking stress. Figures 18 and 19 show the influence of the strain rate on ductility, and Figs. 20 and 21 show the relation between the ductility and the initial breaking stress. The most important ductility value is the value corresponding to the beginning of fracture (technical cohesion limit), not the value corresponding to the end of progressive disintegration. The ductility corresponding to the beginning of fracture will be termed the "true ductility." As the ductility values corresponding to complete fracture are of some interest, however, they are represented in Figs. 18 to 21 both in terms of the total elongation and in terms of the original and final areas of cross-section. In order to represent ductility in terms of the "true strain" (**13**), values of either L/L_0 or A_0/A[5] are plotted as abscissas on a logarithmic scale, and the equivalent extensions are indicated at the top of each figure. The scale used in the representation of true ductility (section (*a*)), however, differs little from a cartesian scale.

Ordinates for all three diagrams of each figure represent values at the beginning of the third stage, unless the stress-strain curve continues to rise as in Fig. 10. For the three specimens represented in Fig. 10, ordinates in Figs. 18 to 21 represent stresses or creep rates at the highest point on the stress-strain curve. The filled-in symbols and open symbols have the same significance in Figs. 18 to 21 as that of the corresponding triangles in Figs. 10 to 16. An open symbol, therefore, indicates that a technical cohesion limit was not reached at the beginning of the third stage, and that the only actual ductility value obtained with that specimen is the value for complete fracture.

The true ductility of monel metal (Fig. 18(*a*)) increases at an increasing rate, with increase in the strain rate. Comparison of the curves in this diagram, moreover, shows that the ductility increases with decrease of temperature. The ductility for complete fracture (diagrams (*b*) and (*c*) of Fig. 18) also increases with increase in the strain rate and with decrease of temperature.

The influence of the strain rate and temperature on the true ductility of copper (Fig. 19(*a*)) is not revealed because the scatter of values is too wide in relation to the slight true ductility of this metal. The true ductility values range irregularly from 0.47 to 0.84 per cent, whereas the values for monel metal range from 0.85 to 7.3 per cent. For complete fracture (Fig. 19 (*b* and *c*)), however, the variation of the ductility of the copper with the strain rate and temperature is similar to the variation of the ductility of monel metal. The ductility of both metals evidently tends to increase with increase in the strain rate and with decrease in temperature.

The difference in true ductility between the monel metal and the copper may be due partly to the difference in the amount of prior cold work. The reductions in section by cold work were 40 per cent and 75 per cent, for the monel metal and copper, respectively.

There is scattered evidence that the ductility continues to increase even at high strain rate (**6**). The *accelerated* increase in the ductility with increase in the strain rate (Figs. 18 and 19), however, would not continue up to a high rate of strain. The curvature would be reversed (Fig. 33). This subject is discussed briefly in a previous paper (**17**).

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[5] L_0 and A_0 are the initial length and area of cross-section, respectively; L and A are the length and area at fracture, either initial or complete.

Figures 20 and 21 show the variation of the ductility with the stress at either the beginning of the third stage or at the highest point of a stress-strain curve such as those in Fig. 10. The ordinates in Figs. 20 and 21 are the same for corresponding points in all three diagrams of each figure. The values for strain rates are given in per cent per 1000 hr., whereas ordinates in Figs. 18 and 19 represent strain rates in per cent per hour.

The variation of the true ductility of monel metal with the technical cohesion limit (for unidirectional tension) may be represented by a single curve (Fig. 20(*a*)). For a constant cohesion limit, therefore, the true ductility is practically unaffected by varying the temperature. Any variation of the temperature is associated with a similar variation of the strain rate, and the two variations have nearly equivalent opposite effects on the ductility. The variation of the true ductility of copper with the technical cohesion limit is clearly revealed in Fig. 21(*a*)). The scatter of results does not obscure the fact that the true ductility of the copper, like that of the monel metal, increases at an increasing rate with increase in the technical cohesion limit.

The ductility for complete fracture of both monel metal and copper, like the true ductility, increases at an increasing rate with increase in the initial breaking stress (Figs. 20 (*b* and *c*) and 21 (*b* and *c*)). The curves for the different temperatures, however, are not blended as they are in diagrams (*a*). The higher the temperature the greater is the plastic deformation during progressive disintegration. A variation of the temperature for a constant technical cohesion limit evidently induces a similar variation of the strain rate, which more than compensates for the effect of the temperature variation on the ductility for complete fracture.

In a study of the influence of the strain rate and temperature on ductility, however, chief attention should be given to the true ductility (Figs. 20(*a*) and 21(*a*)). The ductility of a metal depends on the difference between the initial flow stress and the technical cohesion limit. As the cohesion limit and flow stress vary with the stress system, however, the values to be compared are the flow stress and cohesion limit for the same ratio between the principal stresses. In this paper, however, attention will be confined almost entirely to unidirectional tension, because the stress in a creep test is unidirectional as long as the specimen extends uniformly.

The difference between the technical cohesive strength of a metal and its resistance to plastic deformation is much less than has been generally supposed (**11–19**). After even moderate plastic deformation, the resistance to plastic deformation is only slightly less than the technical cohesive strength (resistance to fracture). This relationship is illustrated by the curves of flow stress (F_0) and cohesion (T_0) in Fig. 17(*a*). The initial technical cohesion limit for unidirectional tension thus is represented approximately by a point a little above the origin of the curve of flow stress. With plastic deformation, the technical cohesive strength increases continuously, at a decreasing rate, but less rapidly than the increase of the flow stress. Fracture under any specific stress combination occurs when the flow stress for that combination becomes equal to the technical cohesion limit. Consequently, for any specific stress combination, the variation of the technical cohesive strength with plastic deformation may be represented qualitatively by a curve starting a little above the origin of the flow-stress curve, rising less rapidly than that curve, and intersecting it at a small angle, at a point representing fracture. For an ordinary tension test, in which the strain rate varies little, both curves rise with decreasing slope. Numerous pairs of corresponding curves of flow stress and cohesion are shown in previous papers (11–19).

Figure 17(*a*) shows the variation of flow stress and technical cohesion limit in a creep test under a constant load during the first two stages. As previously shown (Figs. 10 to 16) the variation of the flow stress (F_0) with extension under constant load is linear. It may be resolved into two oppositely curved variations, the variation due to work-hardening and the variation due to the decreasing strain rate. Work-hardening tends to increase the flow stress at a decreasing rate and the associated decrease in the strain rate tends to decrease the flow stress at a decreasing rate. As the variation of the technical cohesion limit would be expected to be similar to the variation of the flow stress, this variation is represented approximately by the straight line T_0 (Fig. 17(*a*)).

Because of the small angle between the curves of flow stress and cohesion limit (Fig. 17(*a*)) any variable that has even a slight differential effect on these two curves may have a great effect on the ductility. It has been shown that an increase in the radial stress ratio raises the flow stress curve slightly more than it raises the cohesion limit curve and thus decreases the ductility, sometimes very abruptly (11–19), and that lowering the temperature of steels has a similar effect (14, 15, 17). As shown in Figs. 20(*a*) and 21(*a*), however, an increase in the strain rate increases both the cohesion limit and the *ductility*, and a decrease in temperature has a similar effect. The evidence thus indicates that an increase in the strain rate or a decrease in the temperature raises the curve of cohesion limits slightly more than it raises the corresponding curve of flow stress (F_0). This differential effect, though very slight, causes an appreciable increase in the ductility.

INFLUENCE OF TEMPERATURE AND THE STRAIN RATE ON THE FLOW STRESS AND TECHNICAL COHESION LIMIT

For a study of the influence of temperature and the strain rate on the flow stress and technical cohesion limit, diagrams of two types have been prepared, and are shown in Figs. 22 to 26. In each of these figures, ordinates represent either second-stage flow stress (dashed curve) or technical cohesion limit (solid curves). The technical cohesion limit is the "true" stress at the beginning of progressive disintegration.

Figure 22 shows the influence of temperature on the second-stage flow stress and technical cohesion limit of monel metal for various strain rates. Temperatures in degrees Kelvin are represented on a logarithmic scale, as in previous papers, (14 – 18); stresses are represented on a logarithmic scale. The second-stage flow stress values shown in Fig. 22 were obtained from the paper by Bennett and McAdam (3). In that investigation, specimens were tested at various constant temperatures. At each temperature, a specimen was subjected to either an ascending or a descending series of stresses so as to obtain a corresponding series of nearly constant strain rates. At any of these stresses, the test was continued only until the rate became approximately constant. Sometimes an ascending and a descending series of tests were made with the same specimen. The results thus obtained at the same stresses in the two series agree fairly well. Tests with any specimen were discontinued when the strain rate began to increase under constant load. The strain rates given in Fig. 22 are regarded as characteristic values, each corresponding approximately to the value that would be obtained with a single specimen in the second stage of creep.

The curves of technical cohesion limits (Fig. 22) diverge greatly with increase of temperature, as do the curves representing second-stage flow stress. At 600 K., an increase in the strain rate from 1 to 10,000 per cent per 1000 hr. increases the technical cohesion limit by only 20 per cent. At 800 K., the same increase in the strain rate nearly quadruples the cohesion limit. The influence of the strain rate on either the cohesion limit or the flow stress evidently increases greatly with increase in temperature. As might be expected, the curves of cohesion are a little above the corresponding curves representing second-stage flow stress. The evidence shows that the variations of the cohesion limit with temperature and with the strain rate are qualitatively similar to the corresponding variations of the second-stage flow stress.

The curves of flow stress in Fig. 22 have been extended to very low stresses and slow rates. Reversal in the curves would be more pronounced if the ordinary scale of temperatures were used. There is no evidence to show how far the field of technical cohesion limits would extend downward in the field of second-stage flow stresses. However, even if progressive fracture should eventually begin at the lowest stresses and creep rates represented in Fig. 22, the duration of time before incipient fracture would be very great, and a much longer time would elapse before the damage would be apparent.

The variaions of the technical cohesion limit for copper (Fig. 23) with temperature are similar to the variations of the cohesion limit and flow stress for monel metal.

Figure 24 shows the influence of the strain rate on the second-stage flow stress and on the cohesion limit, for monel metal at various temperatures. Both the stress and the strain rate are represented on a logarithmic scale. The variation of the cohesion limit with the strain rate, at a constant temperature, is qualitatively similar to the variation of the second-stage flow stress. The decrease in the slope of each curve with increase in the strain rate signifies a decrease of P (the ratio of d_2S/S to dV/V). For a constant strain rate, P increases with increase in temperature; for constant stress, P decreases with increase in temperature.

Figure 25 is the same as Fig. 24 except that stresses are plotted on a cartesian scale. This method of plotting has been used by some investigators because of some evidence for a linear relationship over a considerable range of stress, although it has been admitted that the relationship does not extend below or above a certain stress range (**5**, **7**). As shown in Fig. 25, however, the results obtained with monel metal give no evidence for a linear relationship, but show merely a minimum curvature near the point of reversal. There are no apparent advantages, and there are some important disadvantages, in the use of this ordinate scale.

Figure 26 shows the variation of the technical cohesion limit of copper with the strain rate, for various temperatures. Stresses in this figure, as in Fig. 24, are plotted on a logarithmic scale. As was concluded from Fig. 23, Fig. 26 shows that the variations of the cohesion limit of copper with the strain rate and temperature are similar to the variations of the cohesion limit of monel metal. The evidence in Figs. 22 to 26, therefore, shows conclusively that the influences of strain rate and temperature on the technical cohesion limit are qualitatively similar to their influences on the flow stress.

The evidence in Figs. 24 and 26 is not in accordance with the prevalent view based on other evidence (4) that the relationship between the stress and the minimum creep rate is logarithmic. Moreover, the curves of flow stress and the curves of cohesion do not appear to follow the generally valuable hyperbolic sine relationship proposed by Nadai and McVetty (23).

RELATION BETWEEN STRAIN RATE AND TEMPERATURE, FOR CONSTANT SECOND-STAGE FLOW STRESS AND CONSTANT COHESION LIMIT

As shown in Figs. 22 to 26, an increase of the strain rate or a decrease in temperature tends to increase both the flow stress and the technical cohesion limit. This implies that the flow stress and the cohesion limit would remain unchanged if the strain rate and the temperature were both increased by equivalent amounts. The equivalence of variations of the strain rate and temperature may be studied by the use of diagrams with strain rates and temperatures as coordinates. Diagrams of this type are shown in Figs. 27 and 28. In both figures, strain rates are represented on the same logarithmic scale, but the temperature scales of the two figures are different. Each curve represents the relation between the strain rate and temperature for the indicated constant stress. As the scatter of experimental points made it impossible to construct separate families of curves to represent the flow stress and cohesion limit of monel metal, a single family of curves has been drawn for monel metal in each figure.

In Fig. 27, the variation in the slope of each curve indicates that the equivalence ratio is not constant (for this temperature scale), but increases with increase in the strain rate and temperature. The divergence of the curves also indicates that this ratio increases with increase in temperature.

Formulas implying that the equivalence ratio is constant, for a given stress and plastic strain, have been proposed by several investigators. A dynamic theory of the strength of metals was proposed in 1925 by Becker (1, 2) and later elaborated by Orowan (24–28). The Becker formula in its simplest form is:

$$V = Ce^{-\frac{A}{kT}}$$

where:

V = the strain rate,
T = the absolute temperature,
A = an energy value corresponding to the heat of activation,
C = a constant, and
k = a constant.

More recently, Zener and Hollomon proposed essentially the same formula, which is here given with some changes of letters (9, 31). They state that strain rate and temperature affect the flow stress only through a single parameter of the form:

$$P = Ve^{\frac{Q}{RT}}$$

where:

Q = a heat of activation, and
R = the gas constant.

A more elaborate formula has been proposed by Kauzmann (10). Still more recently, Dushman, Dunbar, and Huthsteiner (7) used a formula of the same general form, and presented evidence purporting to support the theory of a single parameter connecting the strain rate and temperature.

According to these formulas, a linear relationship should be obtained when the logarithms of the strain rates are plotted against the reciprocals of the "absolute" temperature. This method of representation, therefore, has been used in Fig. 28. Ordinates represent values of log V_M and abscissas represent values of $1000/T$. The curvature and divergence of the lines for the various constant stresses, however, are almost as great in Fig. 28 as in Fig. 27. The evidence in Fig. 28, therefore, gives no support to the view that a single parameter can be used to connect temperature and the strain rate, either for a constant second-stage flow stress or for a constant technical cohesion limit. For monel metal and copper, the equivalence ratio varies with the temperature and with the strain rate, at least within the range of strain rates represented in Fig. 28.

If the curves in Fig. 28 were extended to still lower strain rates, they would become more nearly parallel and probably would become straighter. For a close approach to such a relationship, however, the curves would have to be extended to strain rates much less than 0.001 per cent per 1000 hr. (about 0.01 per cent per year.).

Curves such as those in Fig. 28 should represent stress-temperature-rate relations for a constant amount of plastic deformation. As shown in Figs. 20(*a*) and 21(*a*), the total plastic deformation at any cohesion limit varies little with corresponding variations of the temperature and the strain rate. Because any single curve in Fig. 28 represents a constant stress, therefore, it also represents approximately constant plastic deformation. Any single curve in Fig. 28, therefore, probably is unaffected by a deformation variable, but the heat of activation, Q, possibly varies from curve to curve.

INFLUENCE OF TEMPERATURE AND STRAIN RATE ON THE MECHANICAL PROPERTIES OF COPPER AND MONEL METAL BETWEEN −188 C. AND THE MELTING POINT

It is now possible to correlate the results obtained with copper and monel metal at elevated temperatures with results previously obtained with the same metals at low temperatures (14). The experiments at elevated temperatures were made with the cold-worked copper, N, and monel metal, G; the experiments at low temperatures were made with both the cold-worked and the annealed metals. The correlated results, together with some data obtained by other investigators, are assembled in Figs. 29 to 33. These figures thus show the influence of temperature and the strain rate on the mechanical properties of copper and monel metal between −188 C. (liquid air temperature) and their respective melting points.

Figure 29 shows the influence of temperature and the strain rate on the flow stress, ultimate stress and breaking stress of copper. The yield stress, ultimate stress and breaking stress, determined in ordinary tension tests, vary linearly with temperature (on this scale) between −188 C. and about +100 C. A single line can be used to represent the variation of the breaking stresses of both annealed and cold-worked copper throughout this temperature range. With the help of data obtained by Price (29) with moderately cold-worked copper, the curves for ultimate stress have been extended to the melting point of copper. Above the recrystallization range, the curves for cold-worked copper coincide with the curve for annealed copper. The curves of cohesion at strain rates of 0.2, 10, and 100 per cent per 1000 hr. are derived from Fig. 23.

In the ordinary tension tests the strain rate at the ultimate stress was about 100 per cent per hour. At the yield stress the rate of loading was somewhat slower than is usual in a tension test. At fracture, the local rate of deformation probably was more than ten times the rate at the maximum load. Because of the relatively fast strain rates in the ordinary tension tests, the ductility obtained (75 per cent reduction of area) was much greater than in the creep tests (see Fig. 19). Because of the great ductility obtained in the ordinary tension tests, the effect of plastic deformation on the cohesive strength was sufficient to raise the curve of true breaking stress, R, far above the curves of ultimate stress, U.

At the slow strain rates used in the creep tests the true ductility was only a fraction of one per cent. The cohesion limits, therefore, were only a little above the initial flow stresses. As the *initial* technical cohesive strength (for a specific temperature and strain rate) may be represented by a stress value between the initial flow stress and the beginning of fracture, the cohesion limit obtained in a creep test of copper may be viewed as practically identical with a corresponding *initial* technical cohesion limit. When the strain rate is relatively much faster, as it is in an ordinary tension test, the breaking stress of copper is so far above the yield stress that an index of *initial* technical cohesive strength can be established only approximately. A curve to represent approximately the influence of temperature on the initial technical cohesive strength of cold-rolled copper must be above the yield stress curve (which would be only slightly below the curve of ultimate stress) but below the curve of breaking stress, R. As shown in previous papers (**11–19**) a suitable curve for this purpose should be not far from the curve of ultimate stress. For the cold-rolled copper, N, therefore, the curve of initial technical cohesion, T_0 (Fig. 29) has been placed a little above the corresponding curve of ultimate stress, U. For the annealed copper, N-8, the curve of ultimate stress, U, is far above the curve of yield stress, Y. The curve of initial technical cohesion, T_0, therefore, has been placed a little below the corresponding curve of ultimate stress (**11–19**). Of the two triangular symbols representing creep tests of annealed copper (**4**), one is above the prolonged line of yield stresses. The flow stresses indicated by these symbols, however, are stresses attained after extension to the third stage, whereas the yield stress line represents initial flow stresses. The broken line below the yield stress line Y represents proof stresses corresponding to 0.02 per cent plastic extension. This line points approximately toward zero stress at the melting point (**17**).

Two curves in Fig. 29 show ultimate stress values obtained by Manjoine and Nadai (**20**) with annealed copper at the indicated high strain rates. The strain rates used were about 5000 and 30,000 times the strain rates in an ordinary tension test. Even at temperatures near the melting point, the ultimate stresses obtained at these high strain rates were as high as those obtained in ordinary tension tests by Price (**29**) at temperatures 500 to 700 deg. lower. The weakening effect of the change from the lower to the higher temperature evidently was compensated by the strengthening effect of an equivalent increase in the strain rate. Equivalence ratios, therefore, may be obtained by comparing temperatures and strain rates for points at equal ordinates on the curves for the low and high strain rates. These equivalence ratios may be compared with equivalence ratios obtained from Fig. 28. The comparison supports the evidence in Fig. 28 that the equivalence ratio varies with the temperature and with the strain rate.

In Fig. 30, results of some of the creep tests of cold-drawn monel metal, taken from Fig. 22, are correlated with results previously obtained in ordinary tension tests of cold-drawn and annealed monel metal at lower temperatures (**14**). The curves of ultimate stress for monel metal (Fig. 30), unlike the curves for copper (Fig. 29), do not descend continuously with increase in temperature, but have an upward bulge due to strain aging. The bulge varies in amount and position with the strain rate. The slower the rate, the lower is the temperature at which strain aging has the maximum effect. When strain aging is dominant, a test at a slow rate may give a higher flow stress than a test at a high rate. Although strain aging evidently had some effect in the creep tests made at relatively low temperatures at slow rates, the conclusions derived from these tests are not affected.

With the help of data obtained by Tapsell and Bradley (**30**) the entire course of the curves of ultimate stress of annealed monel metal has been established (Fig. 31). The curve for the cold-drawn monel metal G, if extended, would pass outside the creep test curve for 10,000 per cent per 1000 hr. and would unite with the curve for annealed metal G-14 at a point a little above the recrystallization range. The curves for cohesion of monel metal are similar in significance to the previously discussed curves for copper.

As a basis for a study of the variation of true ductility over a range of temperature from −188 C. almost to the melting point, the results obtained in the creep tests may be correlated with results previously obtained at lower temperatures (**14**). Figure 32 shows the variation of ductility with temperature, for various constant strain rates, and thus is complementary to Figs. 18(*a*) and 19(*a*). Within the field represented in Fig. 32, the ductility increases at an increasing rate with decrease in temperature. For a complete picture of the variation of ductility with temperature and the strain rate, however, it is necessary to include in the view a much wider range of strain rates and temperatures. Such a view for monel metal is shown in Fig. 33. In ordinary tension tests between −188 C. and room temperature, the ductility of the monel metal is high, and varies little with the temperature (**14**). In order that the curves may connect with those representing results of creep tests, the curvature must reverse, as shown in Fig. 33. Evidence obtained from scattered results of tension tests at elevated temperatures indicates that the curves as drawn are qualitatively correct. The additional evidence presented in Fig. 33 thus confirms the previously discussed evidence that the ductility of copper and monel metal increases with decrease in temperature and with increase in the strain rate. The sensitivity to variations of temperature and strain rate decreases with decrease in temperature and with increase in the strain rate.

The great range in ductility represented in Fig. 33 cannot be attributed to the change from intercrystalline to transcrystalline fracture. This change occurs at an equi-cohesion boundary, which depends on both the temperature and the strain rate. At very slow rates, this boundary is far below the ordinary recrystallization temperature. An approximate curve for the equicohesion boundary for copper is shown in Fig. 27(*a*). There may be a reversal of curvature, however, as the curve extends below room temperature. The equicohesion boundary does not imply an abrupt change in the properties on each side of the boundary. The temperatures and strain rates in the creep tests of monel metal (Figs. 3 and 27) were on the intercrystalline side of the boundary, although some of the tests were near the boundary. Most of the creep tests of copper (Figs. 5, 6, 7, and 27) were on the intercrystalline side of the boundary, a few were at the boundary, and a few were on the transcrystalline side.

The wide variations of ductility shown in Fig. 33 are due to a small differential effect of the strain rate and temperature on the flow stress and technical cohesion limit. Because corresponding curves of flow stress and cohesion limits meet at a small angle (Fig. 17(*a*)), any variable that has even a slight differential effect on these curves may have a great effect on the ductility.

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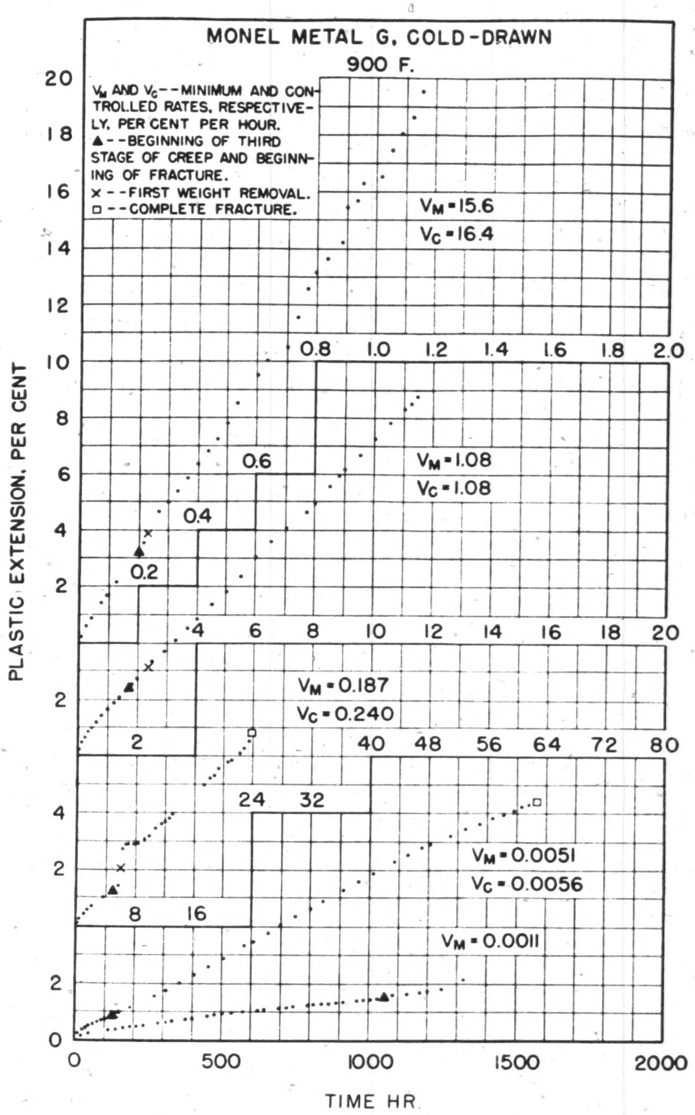
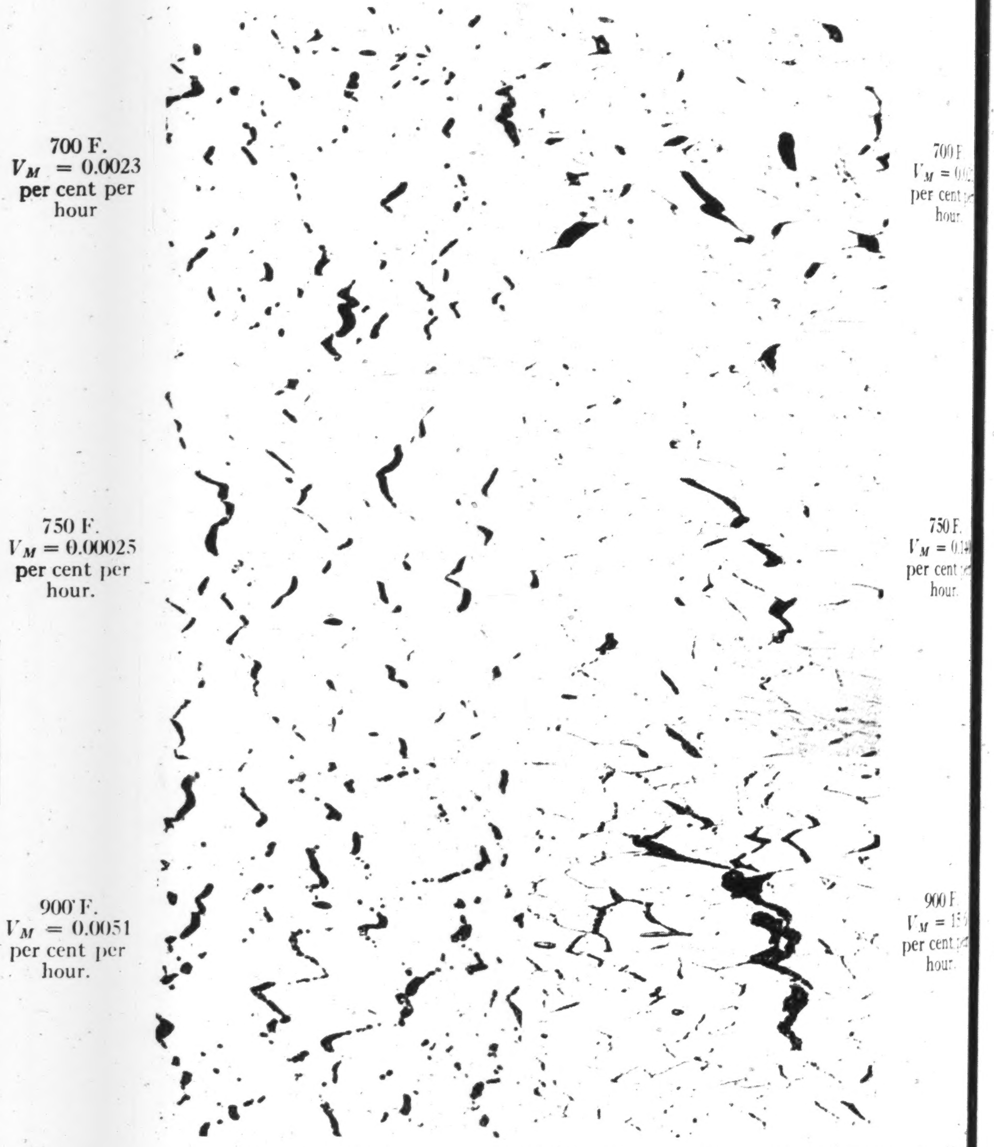


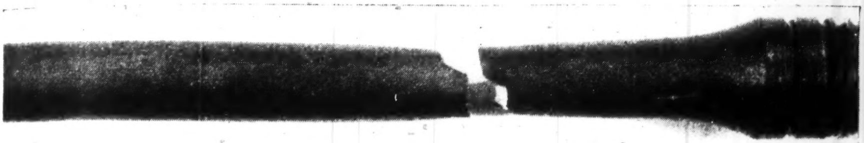
Fig. 1.—Extension-Time Curves for Cold-Drawn Monel Metal.


700 F.
V_M = 0.0023
per cent per
hour
750 F.
V_M = 0.00025
per cent per
hour.
900 F.
V_M = 0.0051
per cent per
hour.
700 F.
V_M = 0.0
per cent
hour.
750 F.
V_M = 0.1
per cent
hour.
900 F.
V_M = 1
per cent
hour.

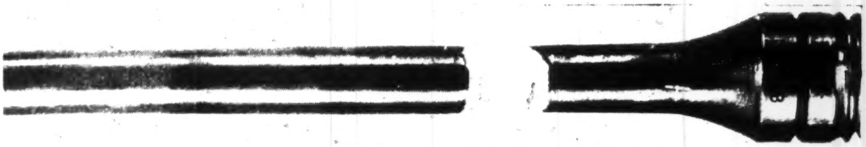
Unetched (× 250).

Etched with a mixture of equal parts of concentrated nitric and glacial acetic acids (× 250).

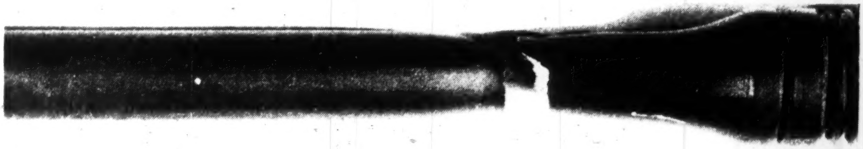
FIG. 2.—Longitudinal Sections After Creep Tests of Cold-Drawn Monel Metal.



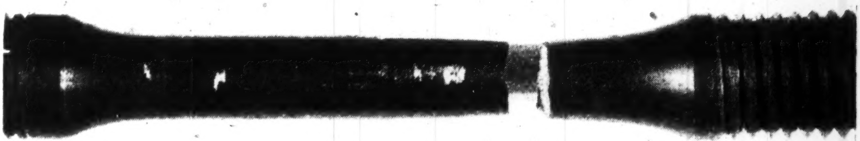
700 F., 93,000 psi., 0.021 per cent, $A_0/A = 1.62$



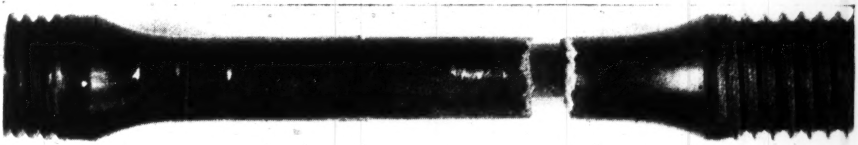
700 F., 90,500 psi., 0.0023 per cent, $A_0/A = 1.17$



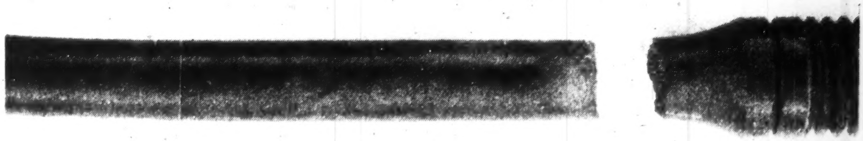
750 F., 92,500 psi., 0.138 per cent, $A_0/A = 1.58$



750 F., 83,100 psi., 0.041 per cent, $A_0/A = 1.29$

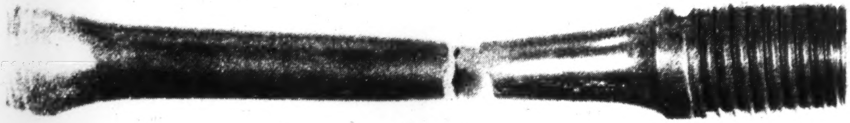


750 F., 72,600 psi., 0.003 per cent, $A_0/A = 1.047$

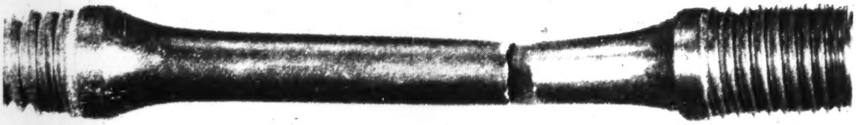


750 F., 64,100 psi., 0.00025 per cent, $A_0/A = 1.026$

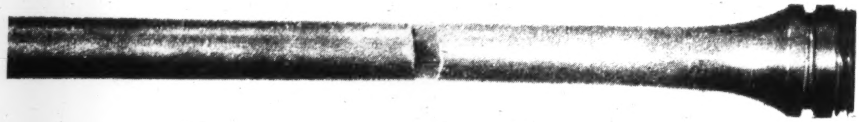
FIG. 3.—Specimens of Cold-Drawn Monel Metal After Creep Tests.



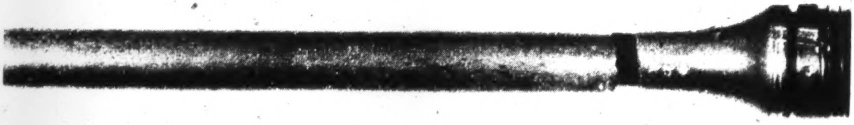
900 F., 78,300 psi., 15.6 per cent, $A_o/A = 1.64$



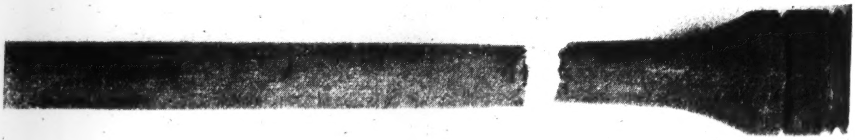
900 F., 68,200 psi., 1.08 per cent $A_o/A = 1.21$



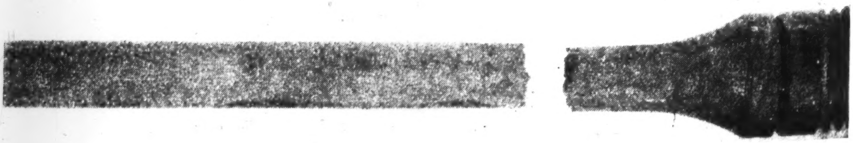
900 F., 58,800 psi., 0.187 per cent, $A_o/A = 1.067$



900 F., 36,500 psi., 0.0051 per cent $A_o/A = 1.05$

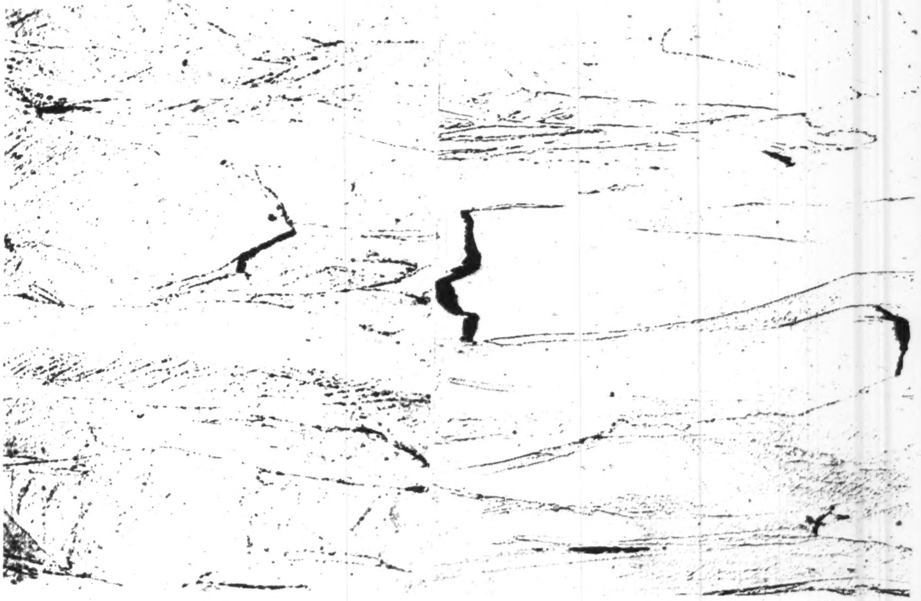


1000 F., 60,500 psi., 12.2 per cent, $A_o/A = 1.35$



1000 F., 50,800 psi., 2.1 per cent, $A_o/A = 1.11$

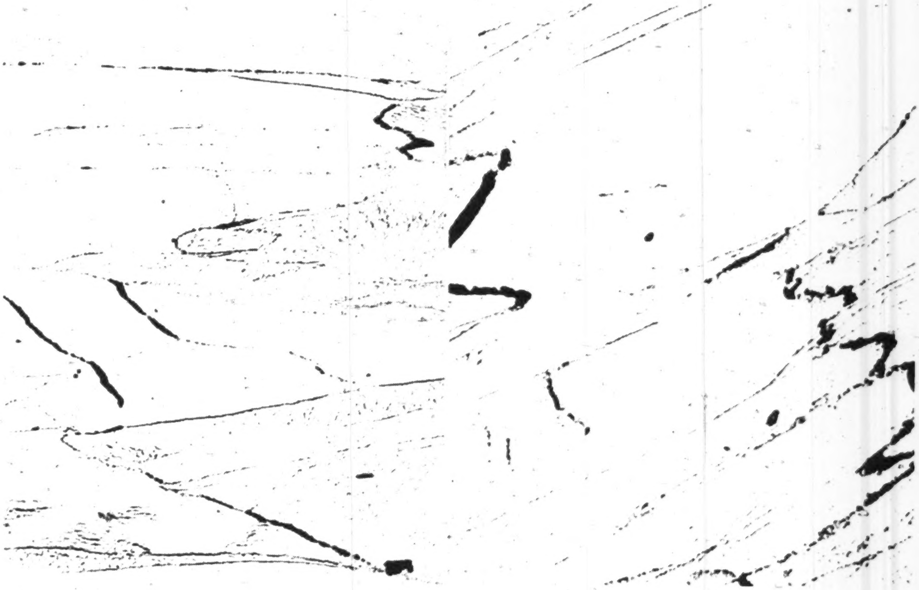
Fig. 4.—Specimens of Cold-Drawn Monel Metal After Creep Tests.



110 F.
$V_M = 0.00028$ per cent per hr.

130 F.
$V_M = 0.00027$ per cent per hr.

FIG. 5.—Longitudinal Sections After Creep Tests of Cold-Rolled Copper.
Etched with a 10 per cent aqueous solution of ammonium persulfate (× 100).



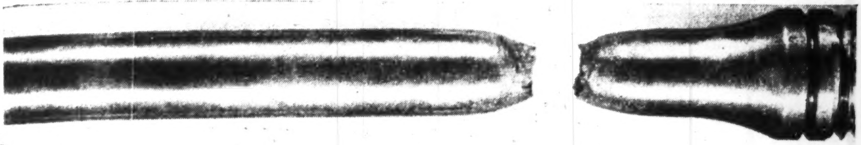
275 F.
$V_M = 0.013$ per cent per hr.

350 F.
$V_M = 0.0011$ per cent per hr.

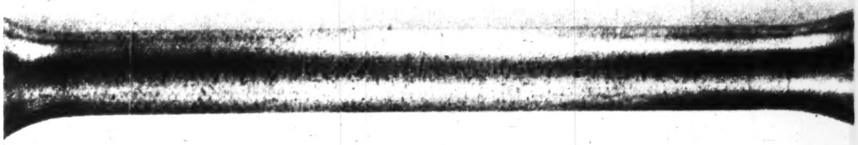
FIG. 6.—Longitudinal Sections After Creep Tests of Cold-Rolled Copper.
Etched with a 10 per cent aqueous solution of ammonium persulfate (× 100).

175 F.
V_M = 0.00017 per cent per hr.
0.25 in. From Fracture
0.04 in. From Fracture
300 F.
V_M = 0.001 per cent per hr.
350 F.
V_M = 0.00021 per cent per hr.

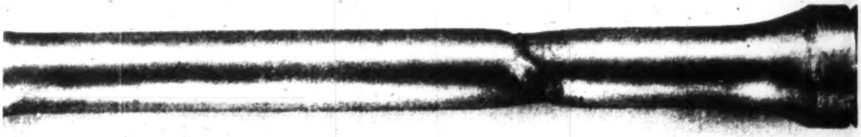
Fig. 7.—Longitudinal Sections After Creep Tests of Cold-Rolled Copper. Unetched (× 50).



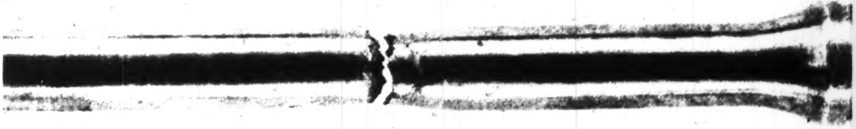
110 F., 52,400 psi., 0.0018 per cent, $A_0/A = 2.8$



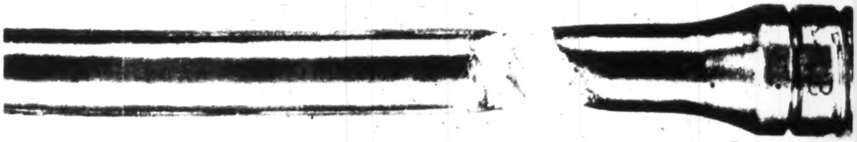
110 F., 52,100 psi., 0.00028 per cent



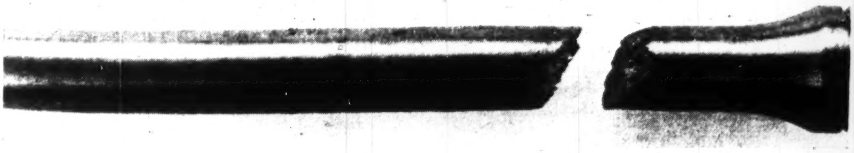
130 F., 51,700 psi., 0.00027 per cent, $A_0/A = 1.37$



175 F., 49,900 psi., 0.00017 per cent

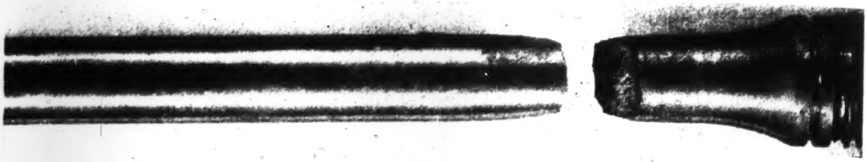


200 F., 48,900 psi., 0.00079 per cent, $A_0/A = 2.09$

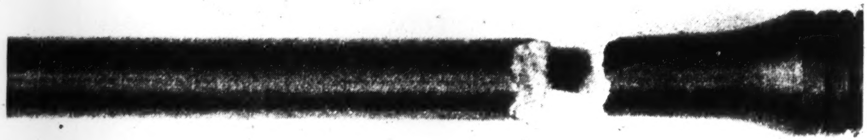


250 F., 47,500 psi., 0.0012 per cent, $A_0/A = 1.12$

Fig. 8.—Specimens of Cold-Rolled Copper After Creep Tests.

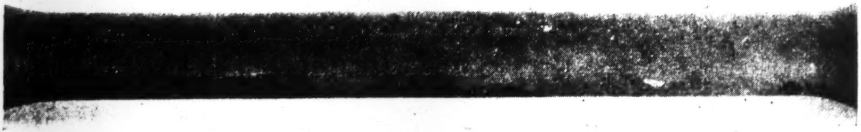


275 F., 47,100 psi., 0.013 per cent, $A_0/A = 1.18$

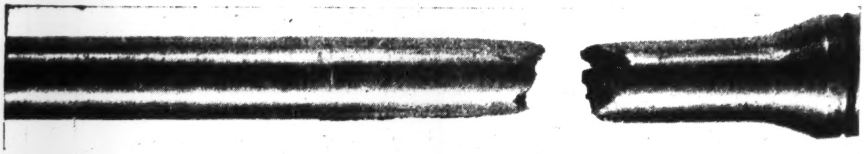


300 F., 43,600 psi., 0.001 per cent, $A_0/A = 1.05$

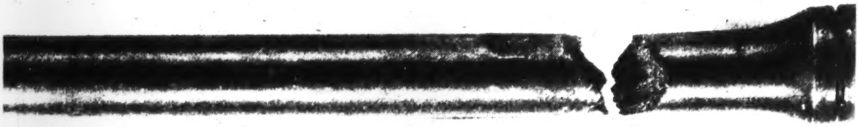
350 F., 40,700 psi., 0.0011 per cent, $A_0/A = 1.13$



350 F., 36,100 psi., 0.00021 per cent



375 F., 45,300 psi., 0.180 per cent, $A_0/A = 1.2$



375 F., 41,900 psi., 0.009 per cent, $A_0/A = 1.14$

FIG. 9.—Specimens of Cold-Rolled Copper After Creep Tests.

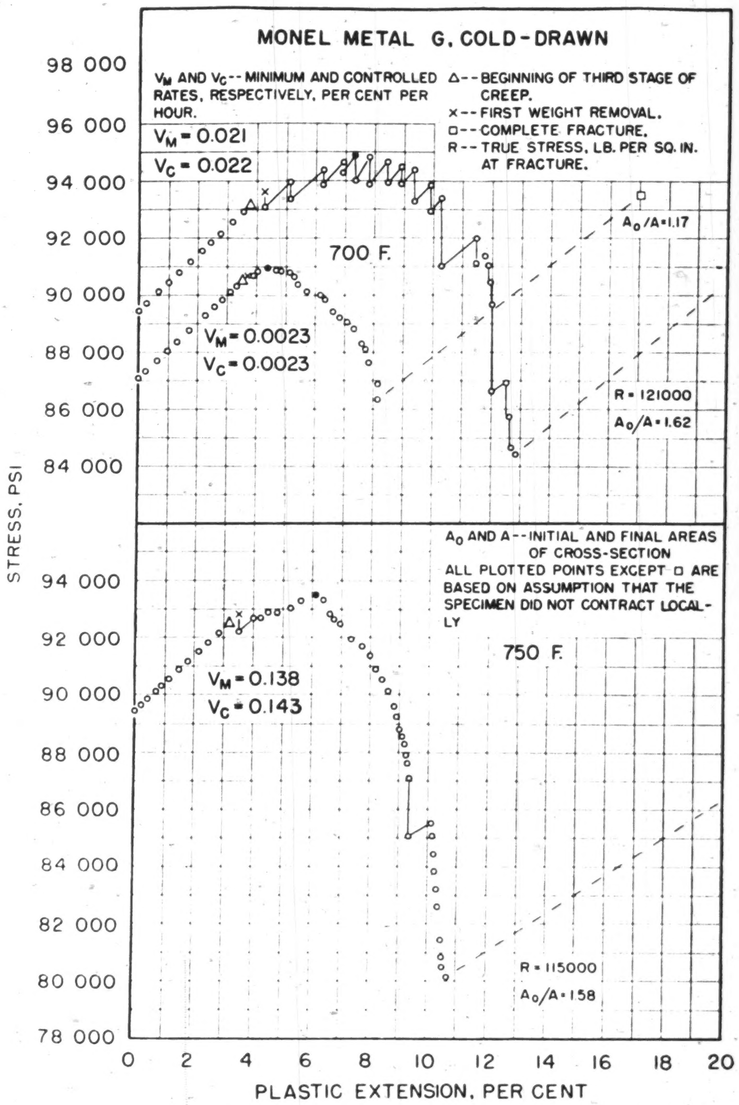


Fig. 10.—Variation of Flow Stress with Extension of Cold-Drawn Monel Metal.

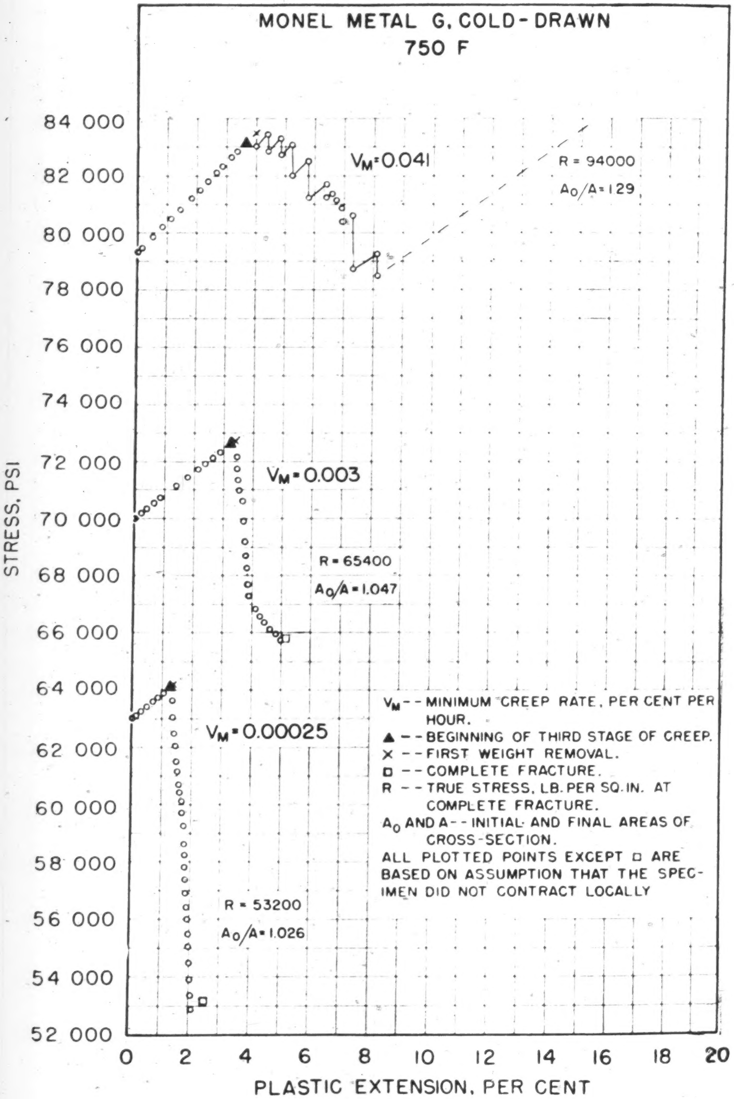


FIG. 11.—Variation of Flow Stress with Extension of Cold-Drawn Monel Metal.

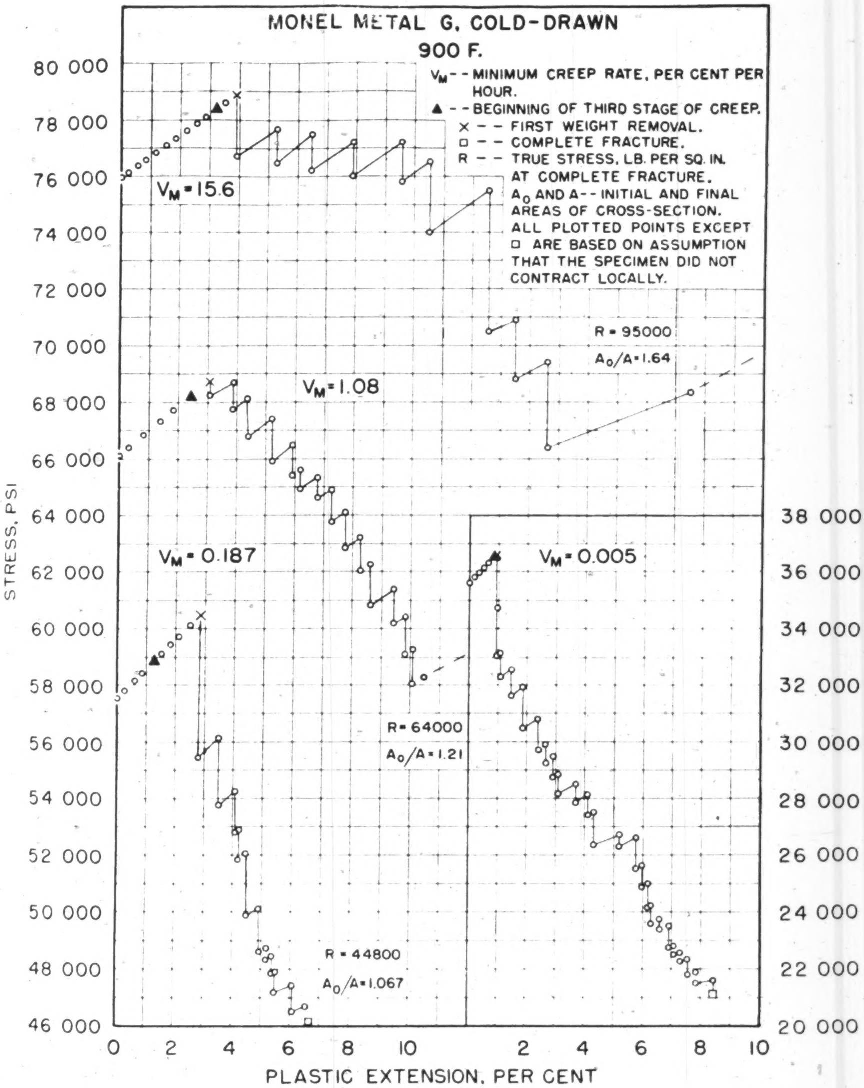


Fig. 12.—Variation of Flow Stress with Extension of Cold-Drawn Monel Metal.

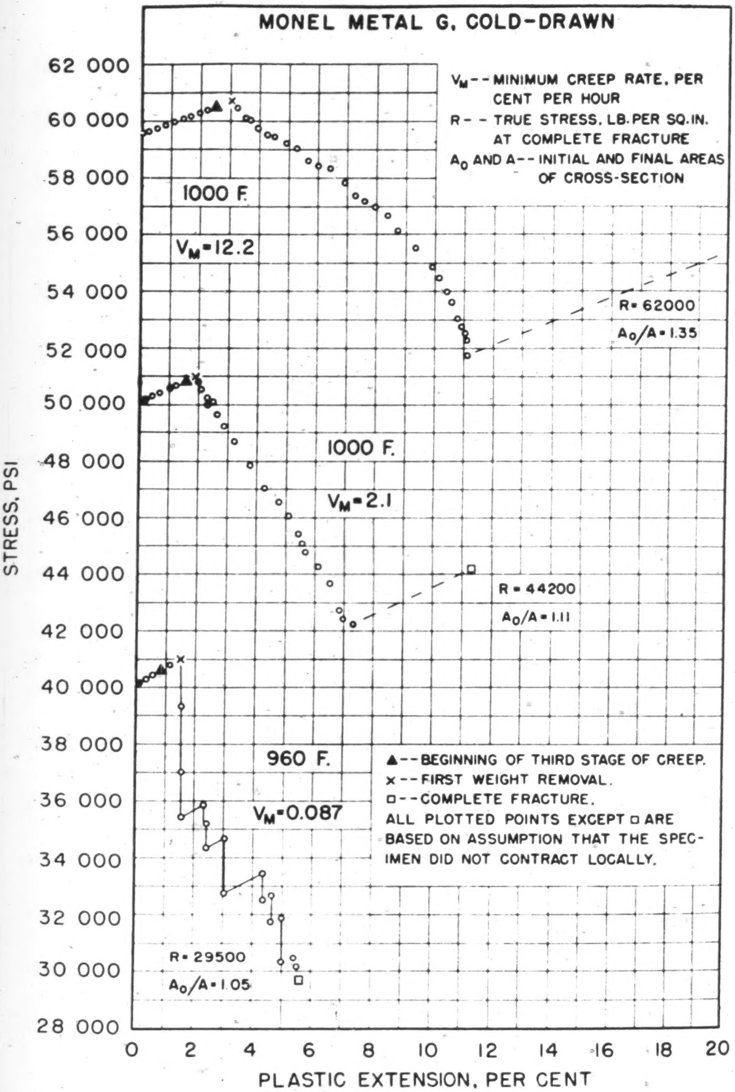


FIG. 13.—Variation of Flow Stress with Extension of Cold-Drawn Monel Metal.

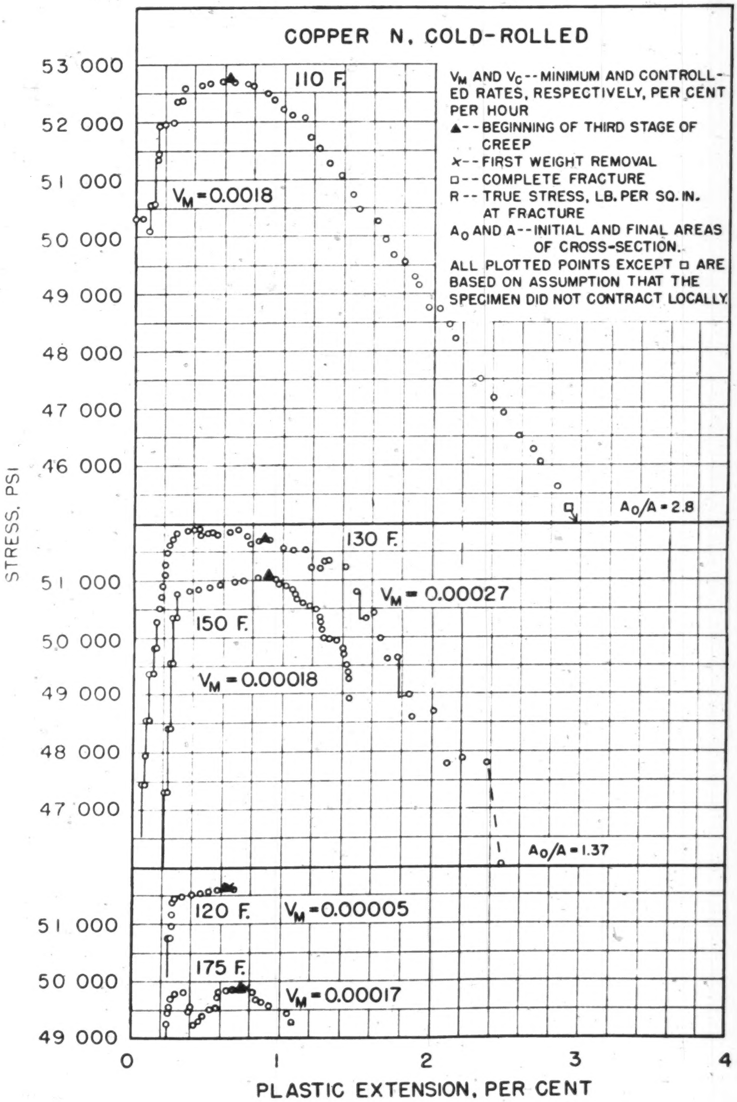


Fig. 14.—Variation of Flow Stress with Extension of Cold-Rolled Copper.

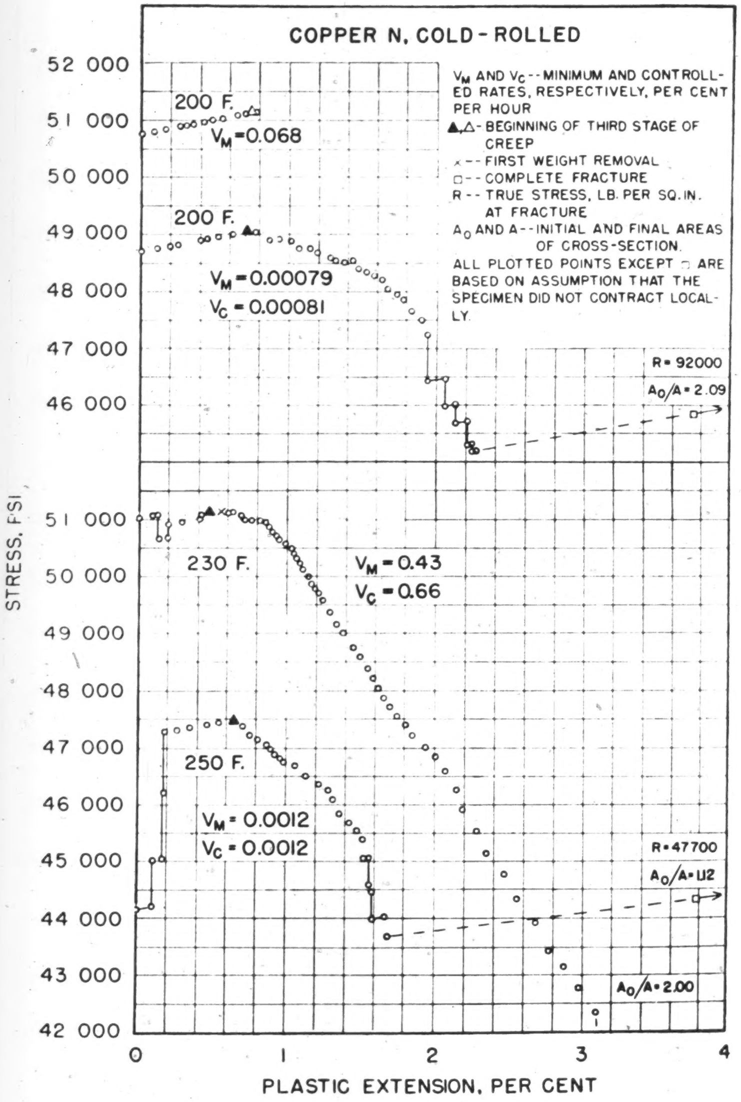


FIG. 15.—Variation of Flow Stress with Extension of Cold-Rolled Copper.

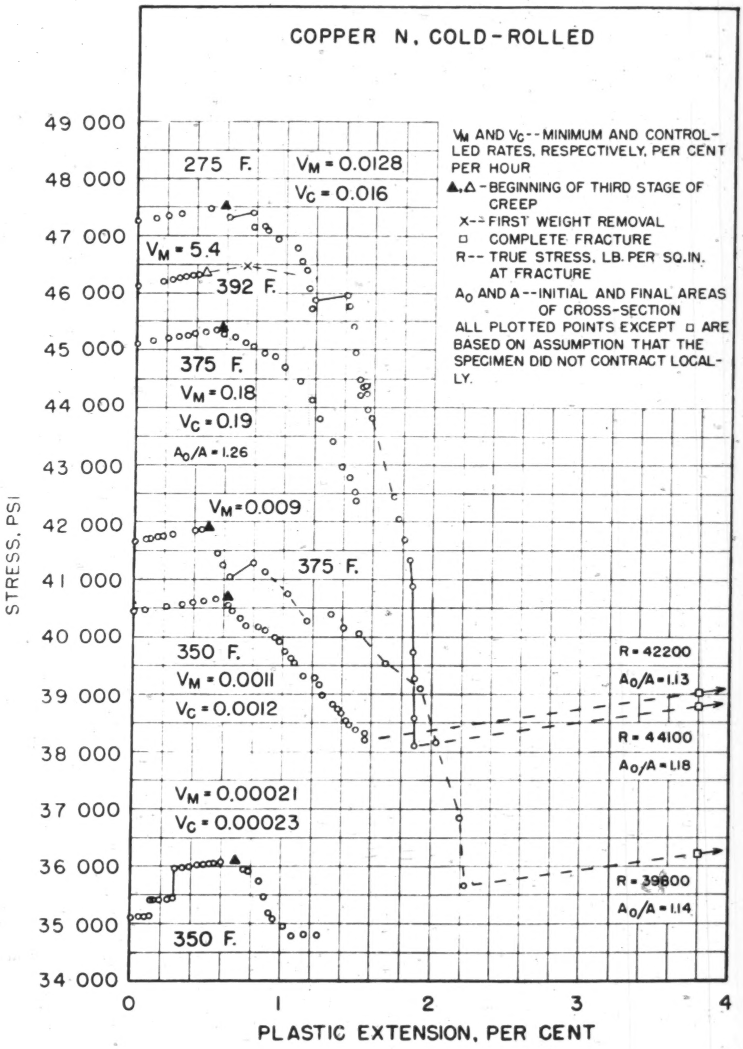


Fig. 16.—Variation of Flow Stress with Extension of Cold-Rolled Copper.

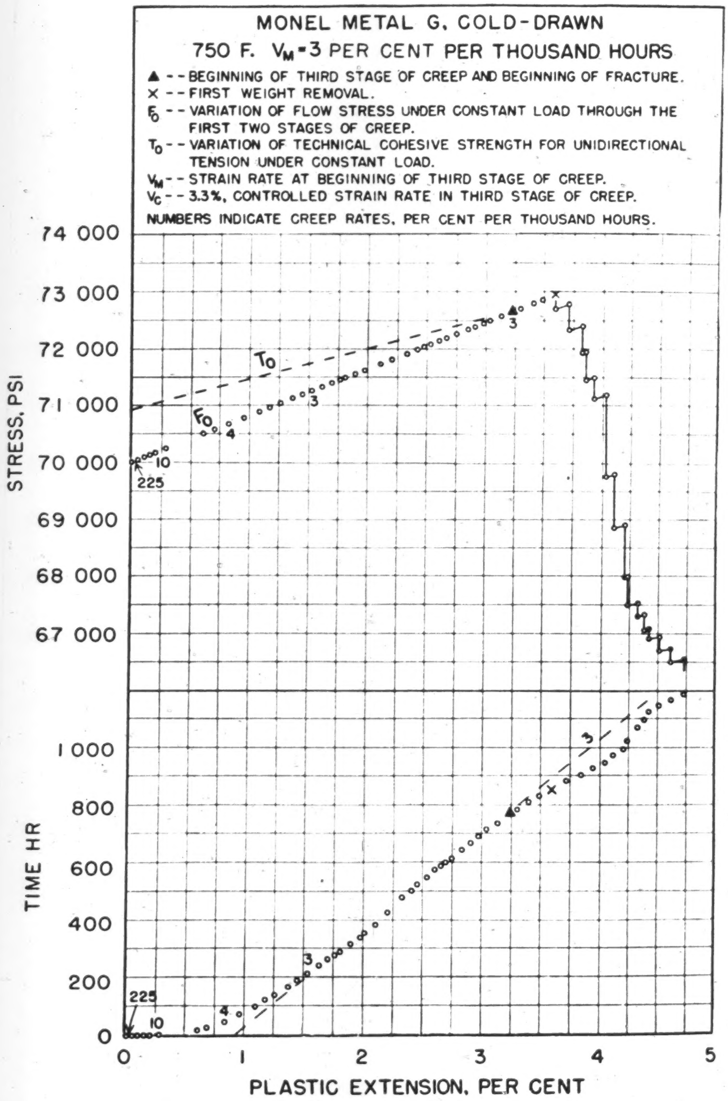


Fig. 17.—Variation of Flow Stress, Creep Rate, and Technical Cohesion Limit with Extension of Cold-Drawn Monel Metal at 750 F.

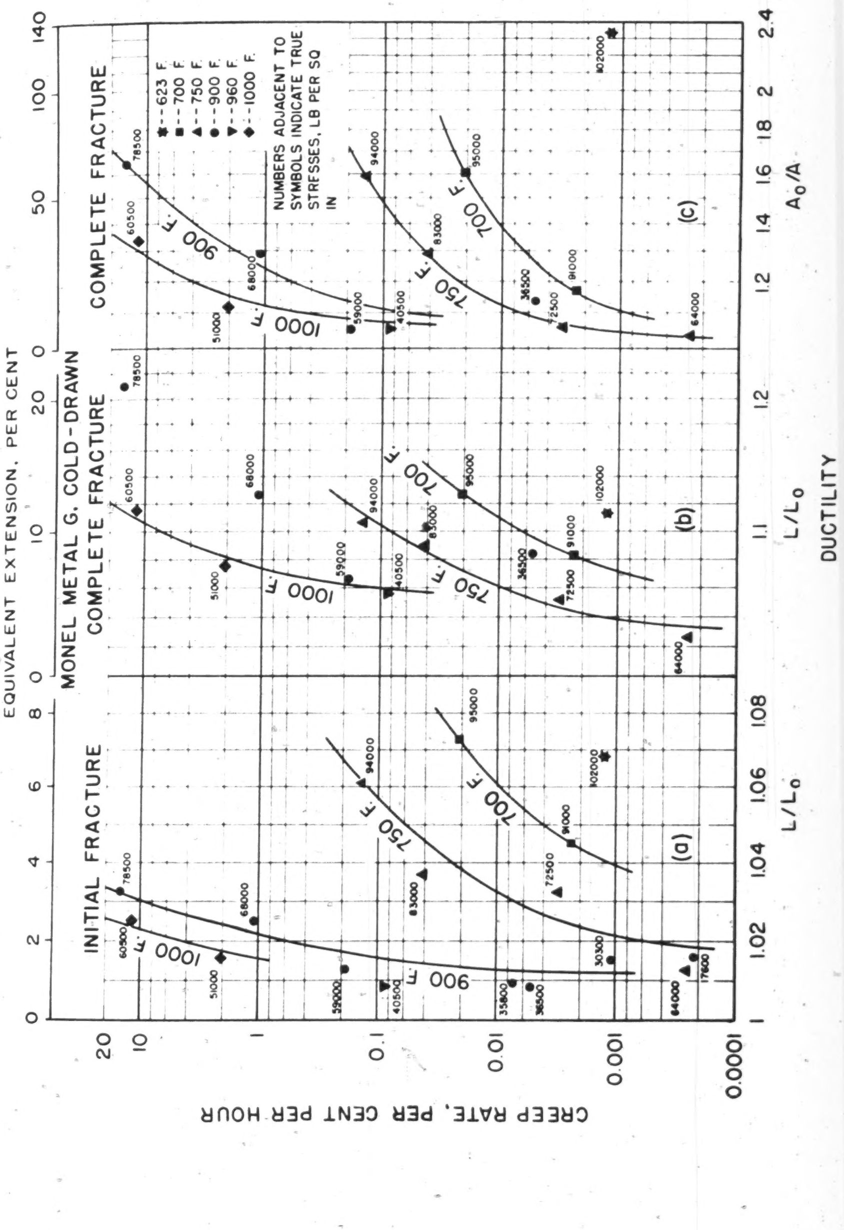


Fig. 18.—Influence of the Strain Rate on the Ductility of Cold-Drawn Monel Metal at Various Temperatures.

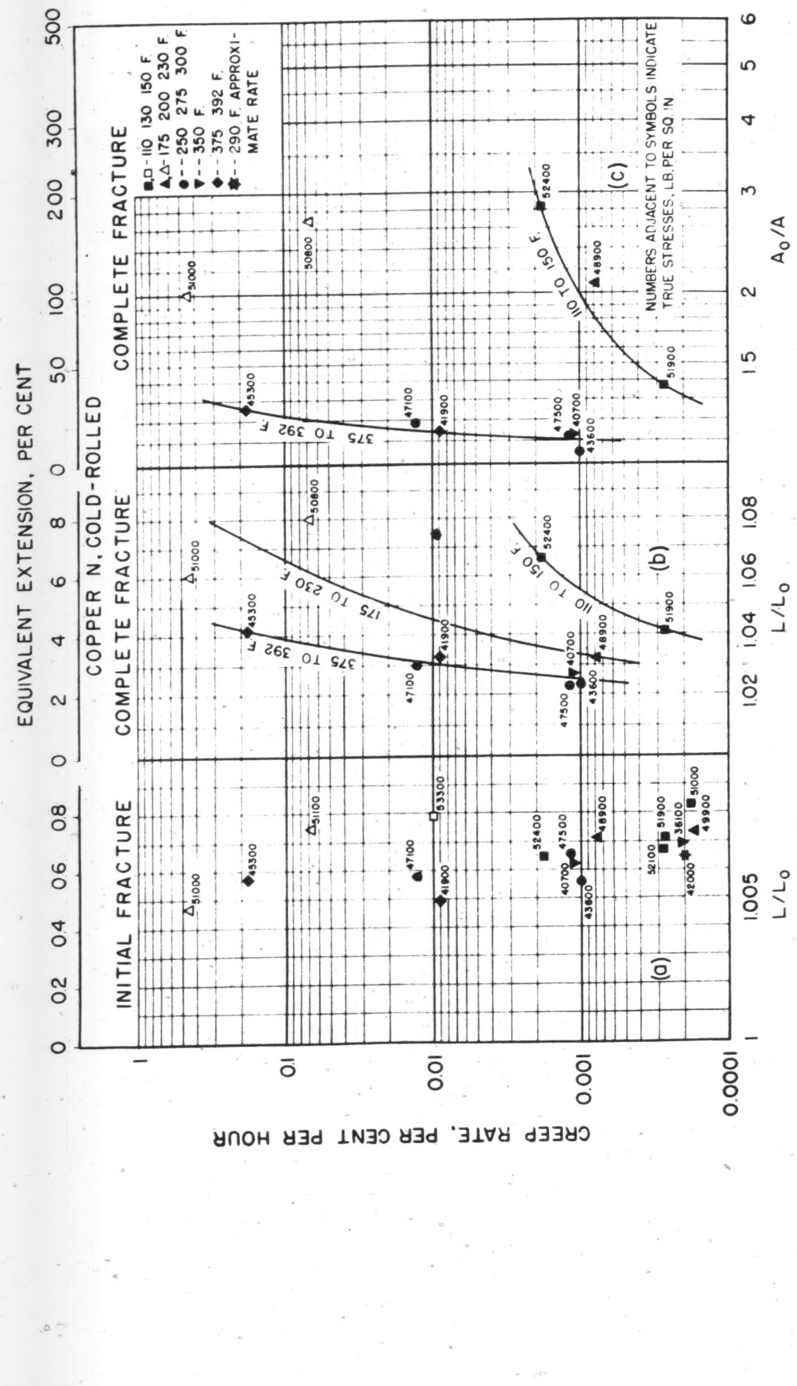


FIG. 19.—Influence of the Strain Rate on the Ductility of Cold-Rolled Copper at Various Temperatures.

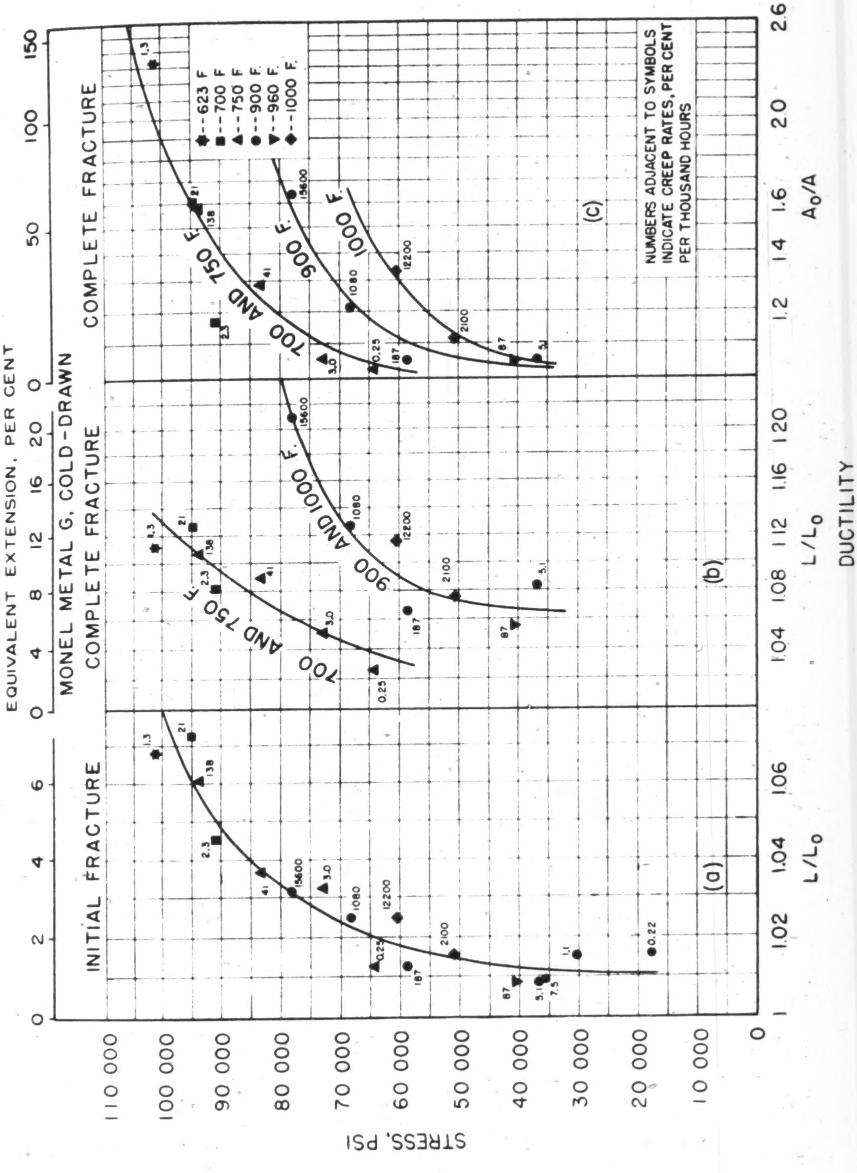


FIG. 20.—Relation Between Initial Breaking Stress and Ductility of Cold-Drawn Monel Metal at Various Temperatures.

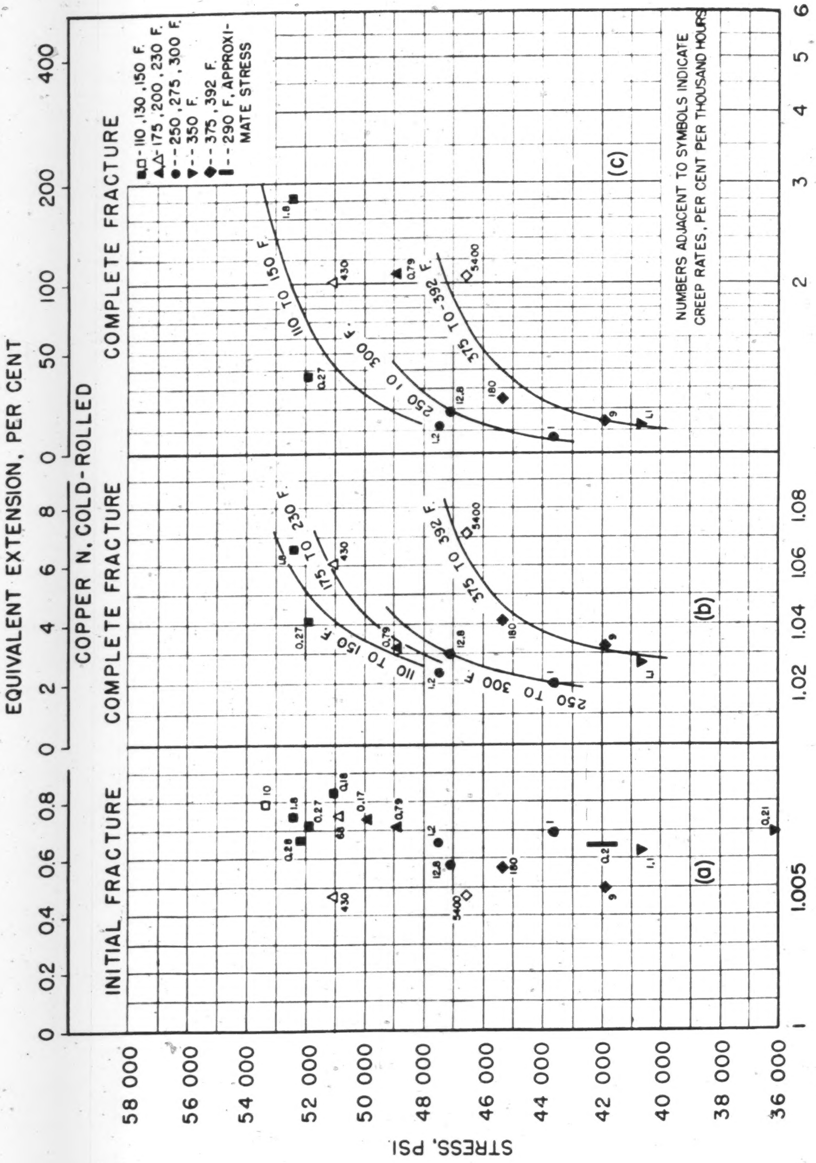


Fig. 21.—Relation Between Initial Breaking Stress and Ductility of Cold-Rolled Copper at Various Temperatures.

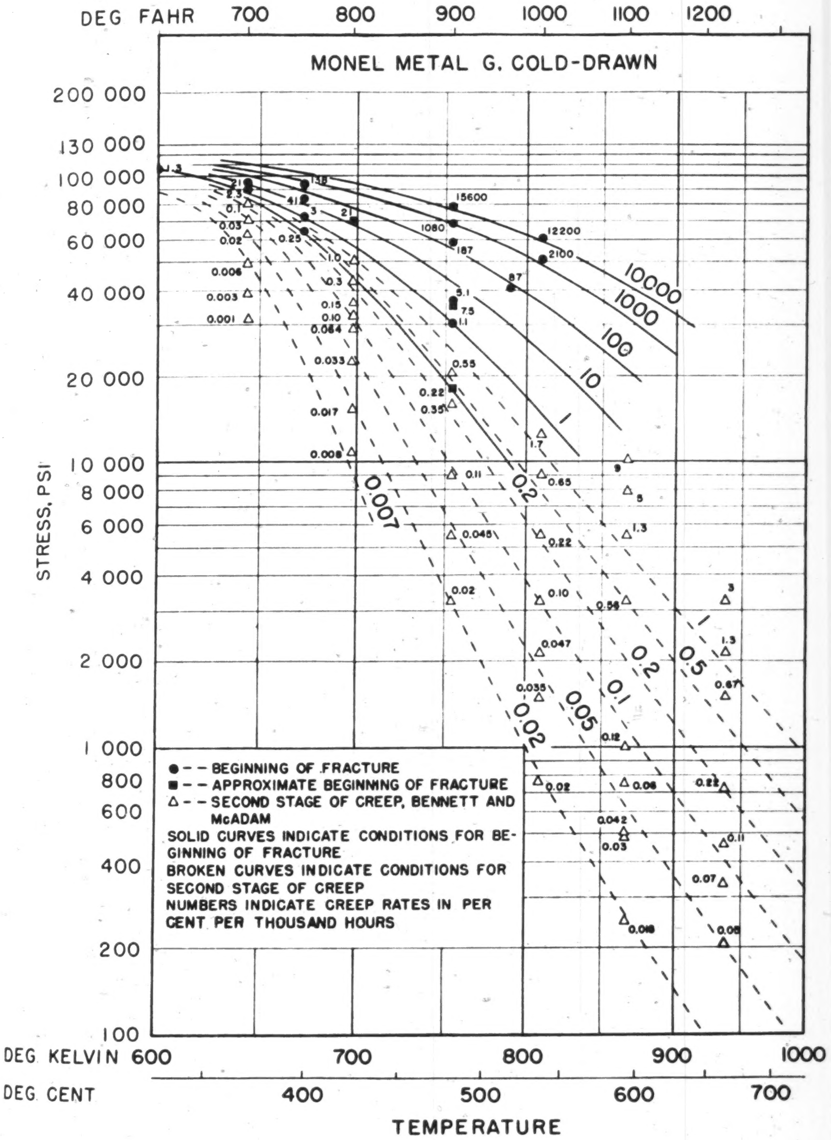


Fig. 22.—Influence of Temperature on the Second-Stage Flow Stress and Initial Breaking Stress of Cold-Drawn Monel Metal at Various Strain Rates.

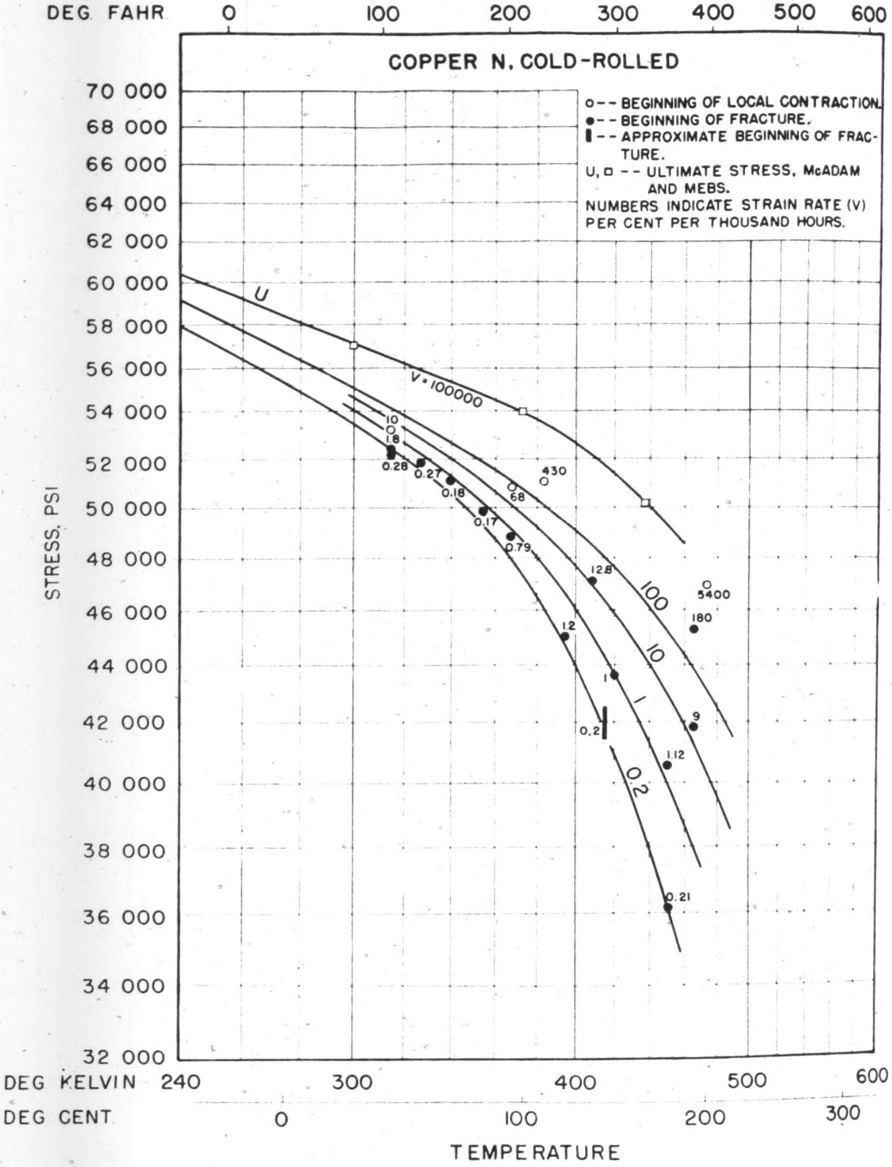


FIG. 23.—Influence of Temperature on the Initial Breaking Stress of Cold-Rolled Copper at Various Strain Rates.

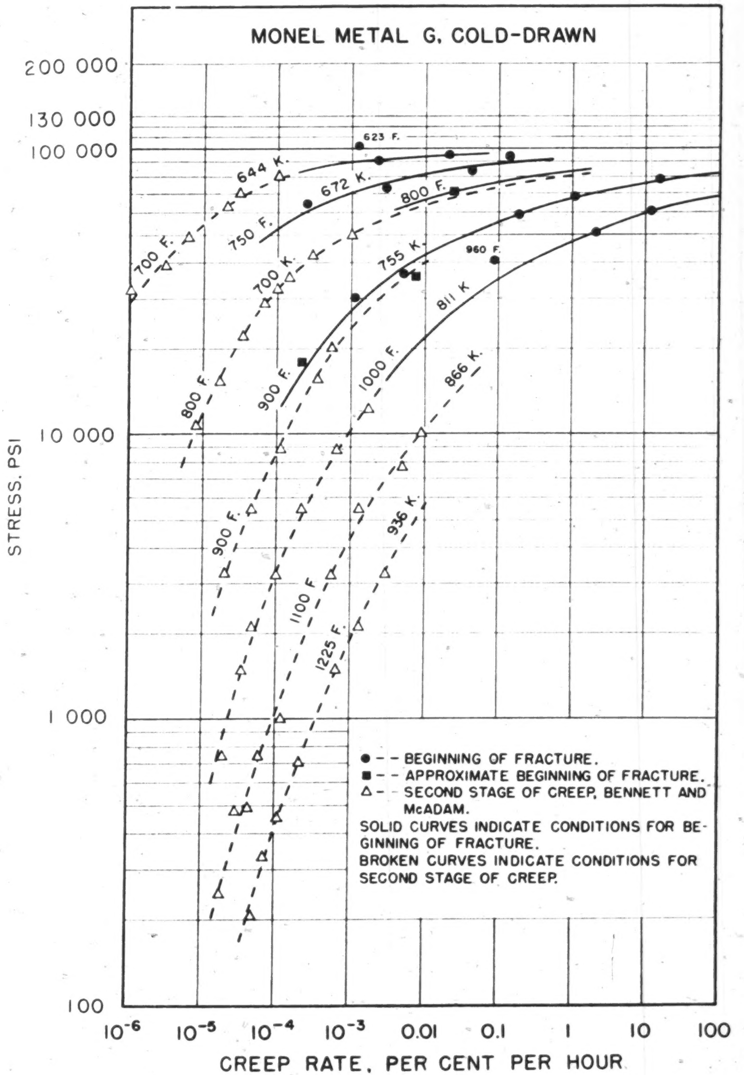


FIG. 24.—Influence of Strain Rate on the Second-Stage Flow Stress and Initial Breaking Stress of Cold-Drawn Monel Metal at Various Temperatures.

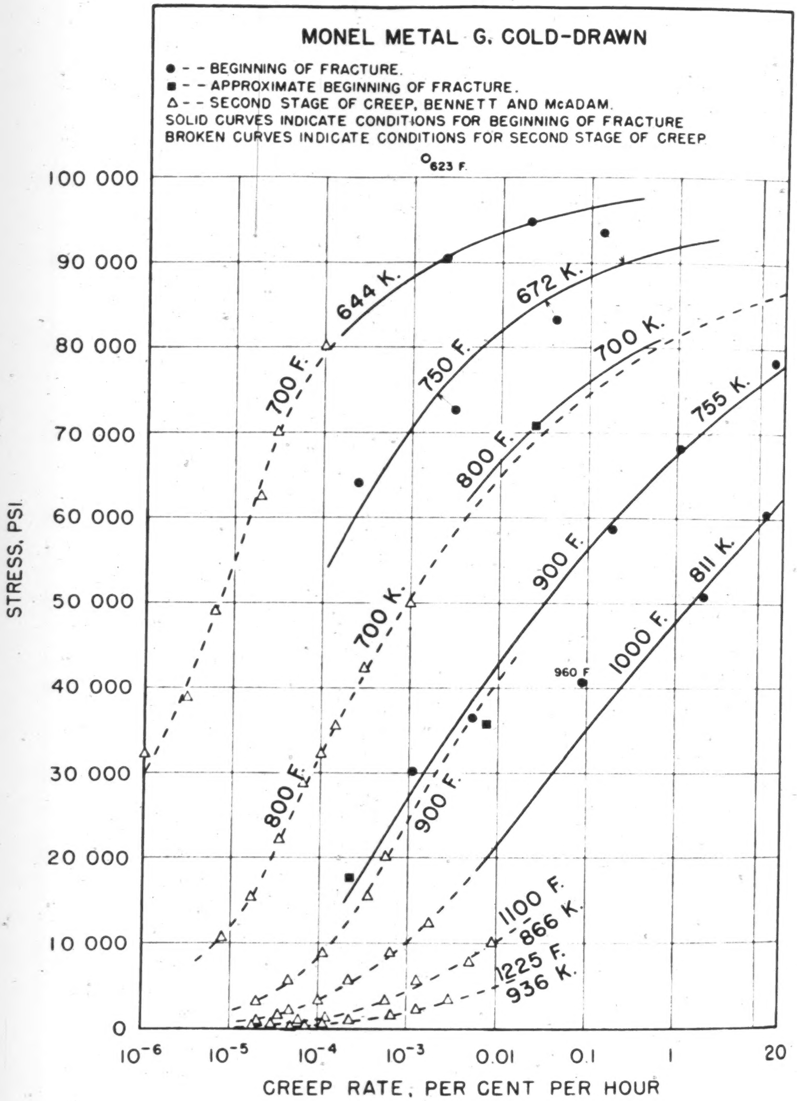


FIG. 25.—Influence of the Strain Rate on the Second-Stage Flow Stress and Initial Breaking Stress of Cold-Drawn Monel Metal at Various Temperatures.

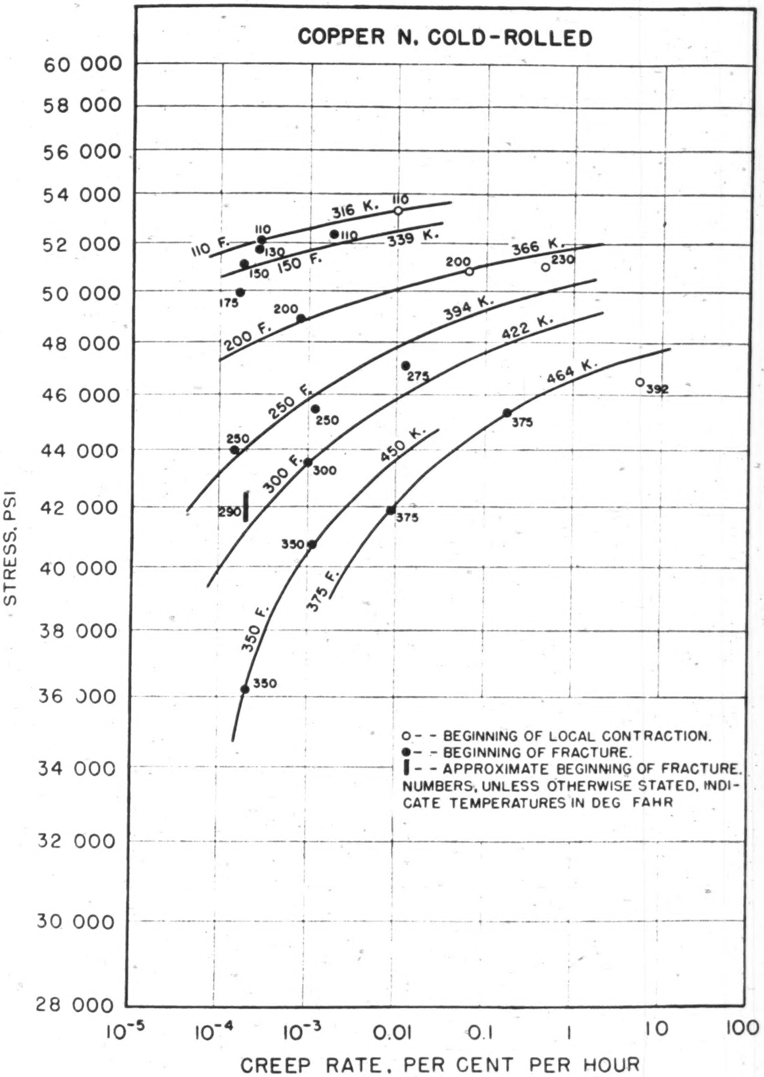


FIG. 26.—Influence of the Strain Rate on the Initial Breaking Stress of Cold-Rolled Copper at Various Temperatures.

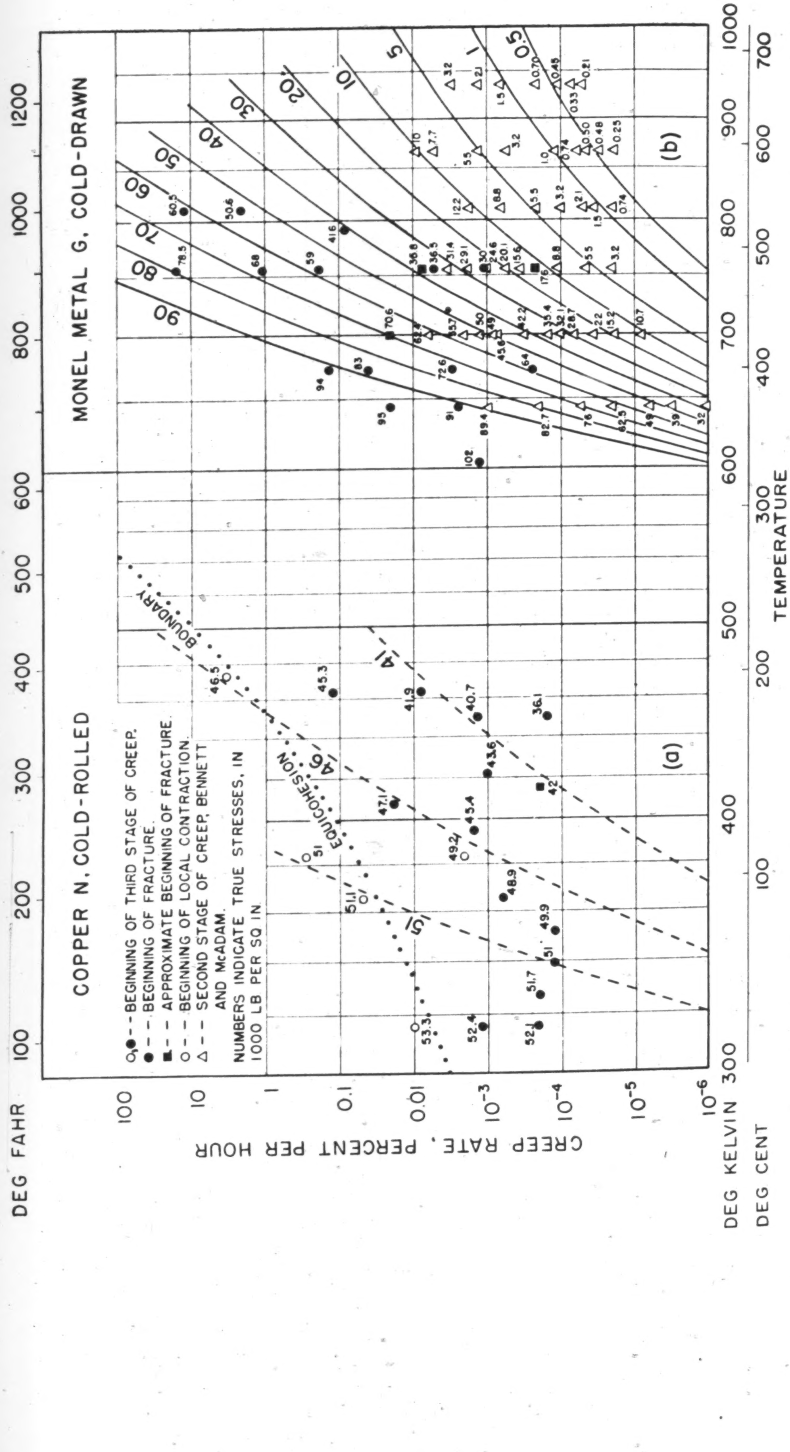


FIG. 27.—Variation of the Strain Rate with Temperature for Various Second-Stage Flow and Initial Breaking Stresses.

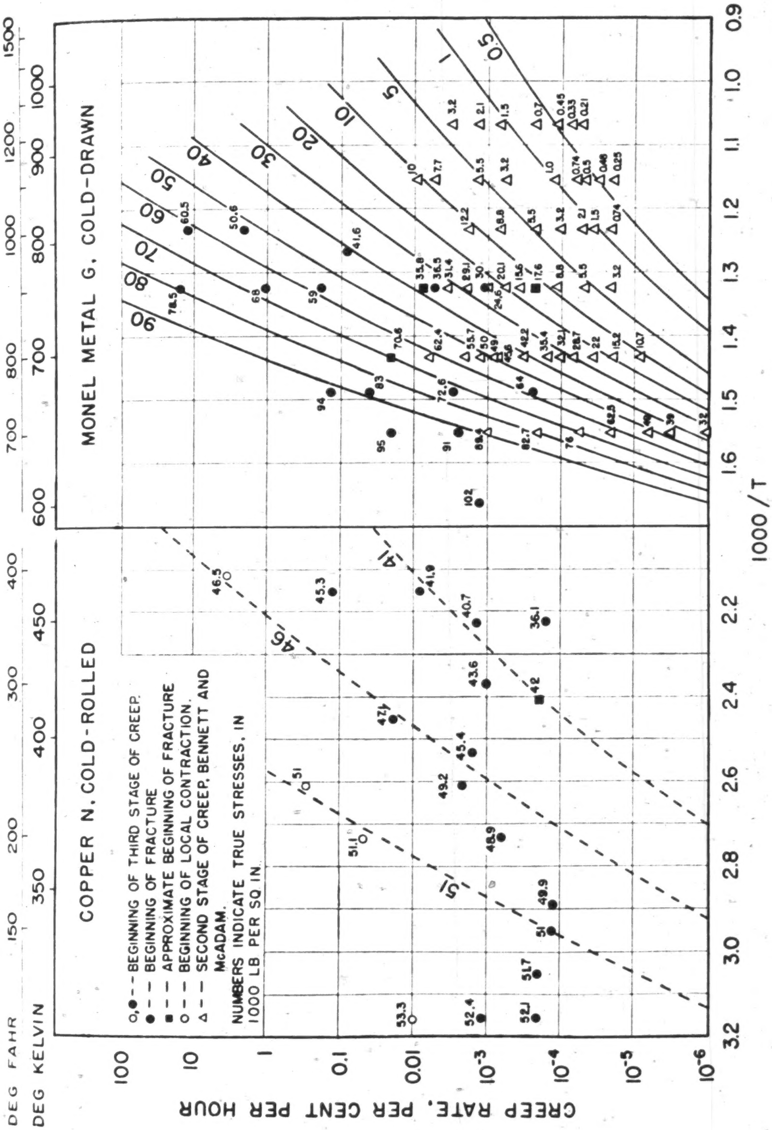


FIG. 28.—Variation of the Strain Rate with the Reciprocal of the degs. Kelvin for Various Second Stage Flow and Initial Breaking Stresses.

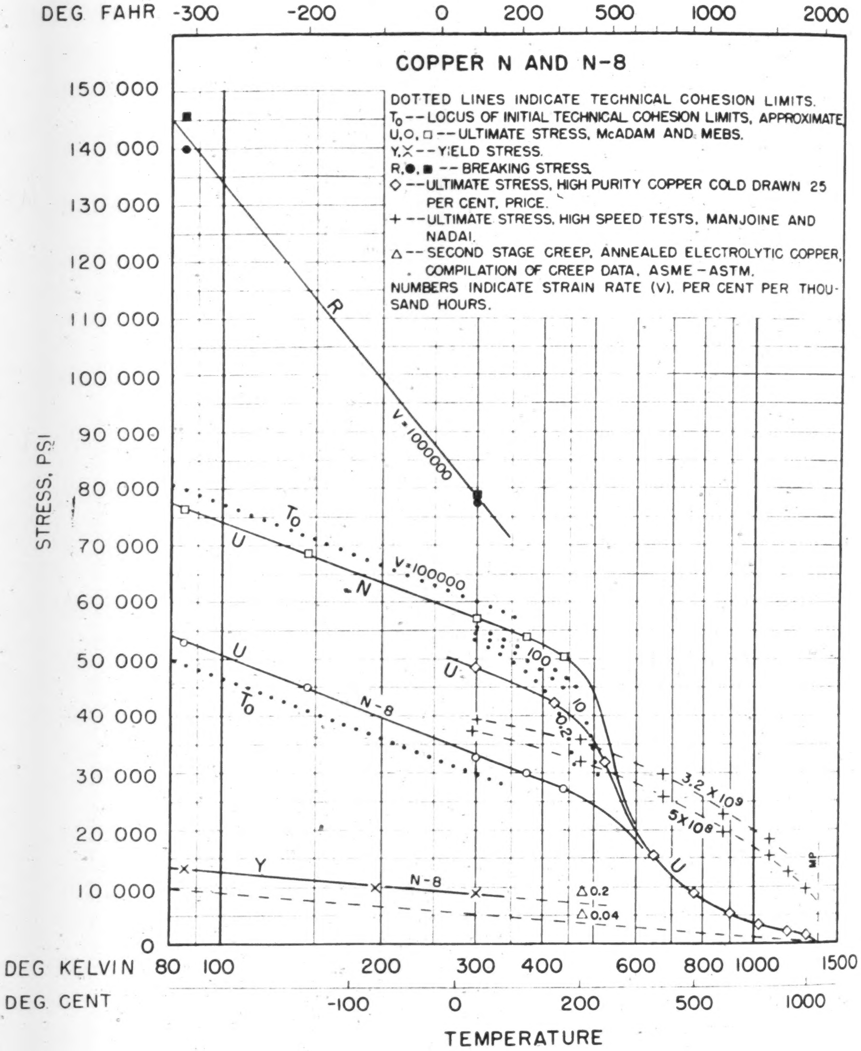


FIG. 29.—Influence of Temperature on the Strength of Copper for Various Strain Rates.

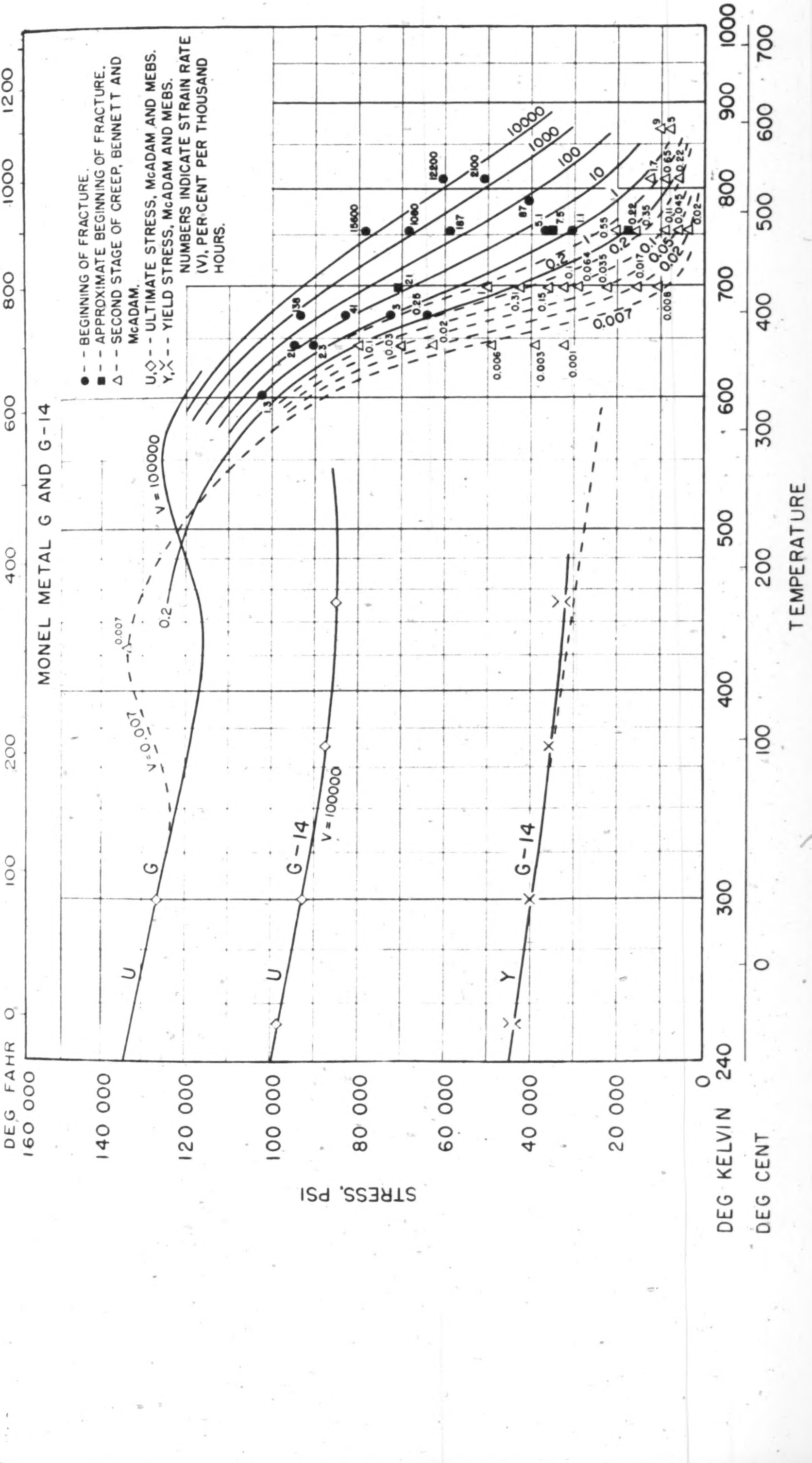


FIG. 30.—Influence of Temperature on the Strength of Monel Metal for Various Strain Rates.

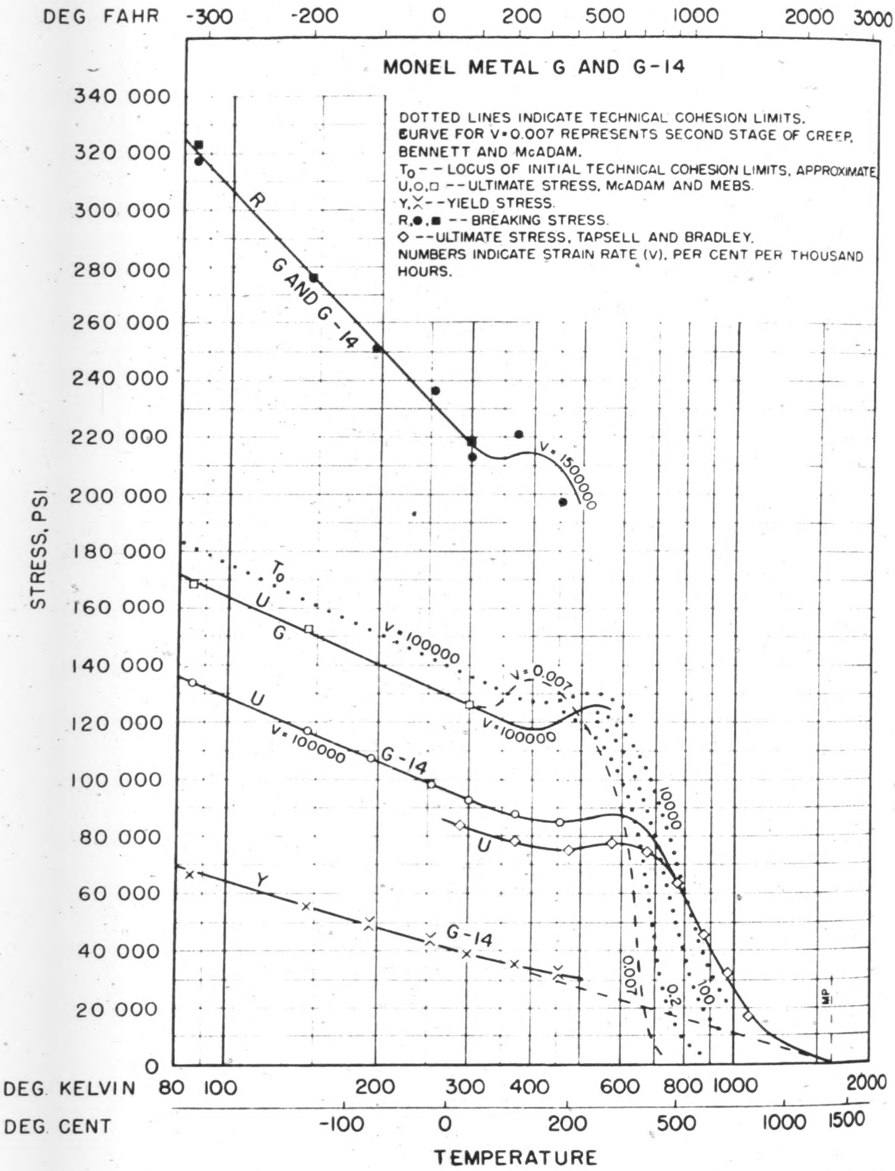


Fig. 31.—Influence of Temperature on the Strength of Monel Metal for Various Strain Rates.

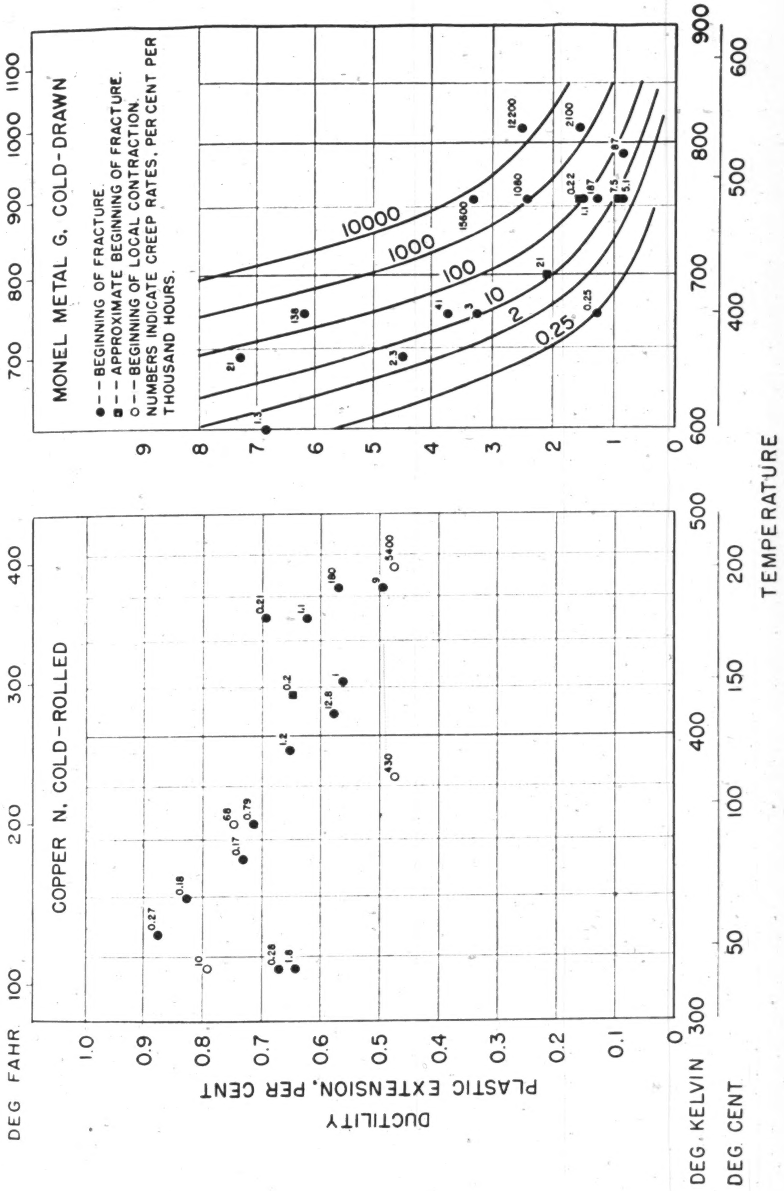


Fig. 32.—Influence of Temperature on the Ductility of Monel Metal and Copper at Various Strain Rates.

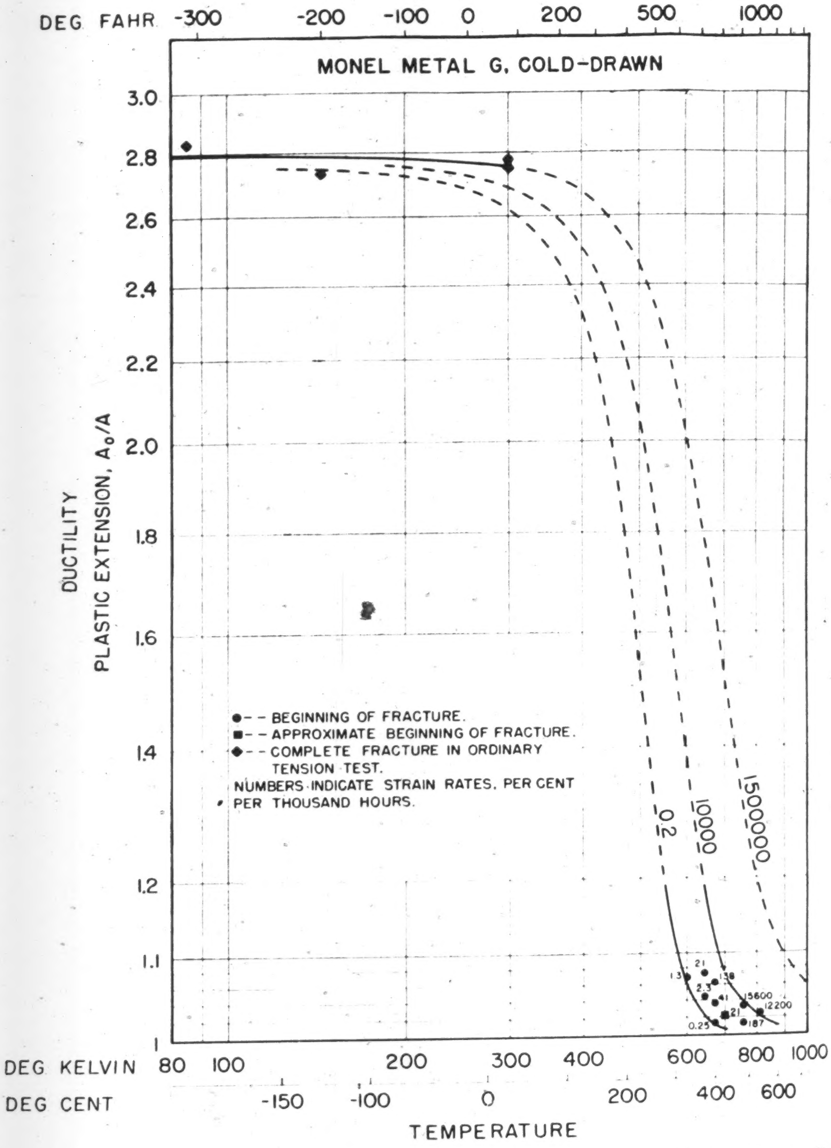


Fig. 33.—Influence of Temperature on the Ductility of Monel Metal at Various Strain Rates.

COMPRESSIVE PROPERTIES OF ALUMINUM ALLOY SHEET AT ELEVATED TEMPERATURES*

By Alan E Flanigan,[1] Leslie F. Tedsen,[1] and John E. Dorn[1]

Synopsis

Apparatus has been developed to allow the compression testing of sheet materials at elevated temperatures. It includes a testing fixture, a special extensometer, and an oil-bath furnace which are described in detail. The short-time compressive properties of five high-strength aluminum alloy sheet materials have been determined at temperatures up to 300 F. after exposures ranging from ½ to 1000 hr. The materials include 24S-T, 24S-T81, 24S-T86, R301-T, and 75S-T. The effects of temperature and time on compressive yield strength are compared with those for the corresponding tensile properties and it is found that approximate values of compressive yield strength may be derived from calculations based upon the tension values. Of the materials tested, the members of the 24S-T8 series appear to offer the best possibilities for elevated-temperature service.

The compressive properties of aluminum alloy sheet are of particular interest in the aircraft industry and in other fields where the design of structures requires consideration of buckling. Recently techniques have been developed for the compression testing of sheet materials at room temperature. Little if any attention, however, has been devoted to the determination of elevated-temperature compressive properties. This paper describes apparatus developed for use in short-time compression tests at elevated temperatures and in addition presents the results obtained on five high-strength aluminum alloy sheet materials at temperatures up to 300 F. The work was done at the University of California as a portion of the restricted project NRC-548. It was financed by the War Production Board and was supervised by the Office of Production Research and Development. This paper has been released for publication by the latter agency.

Precipitation Hardening

As is well known, the high-strength aluminum alloys achieve their high values of tensile and compressive yield strength as a result of precipitation hardening (**1**, **2**).[2] In production they are subjected to a solution heat treatment followed by "aging" (precipitation hardening) either at room temperature or at suitable elevated temperatures. A considerable increase in yield strength results from room-temperature precipitation ("natural aging"), and through the elevated-temperature process ("artificial aging") it is possible to obtain even higher values. During the first stages of artificial aging the yield strength may decrease slightly, but subsequently it increases until it reaches a maximum value, in which condition the material is said to be "fully aged." The higher the temperature, the more rapidly is the fully aged condition obtained. In the case of the alloy 24S the yield strength attainable is influenced by the application of cold work preceding artificial aging. The greater the degree of deformation the greater is the yield strength attainable. After full aging, further exposure results in "overaging" attended by a steady decrease in yield strength. In the production of the artificially aged materials, temperatures and times are selected to achieve full aging.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

[1] Research Engineer, Engineer, and Associate Professor, respectively, University of California, Berkeley, Calif.

[2] Boldface numbers in parentheses refer to the list of references appearing at the end of this paper, see p. 967.

In view of the preceding remarks it is clear that a study of the elevated-temperature properties of such materials requires consideration of the effects of time at temperature (exposure time) as well as temperature.

For the purpose of this paper it is convenient to classify the high-strength materials as follows:

Group A.—Naturally aged materials. Precipitation-hardenable alloys which have been solution heat treated and aged at room temperature.

Group B.—Artificially aged materials. Precipitation-hardenable alloys which have been solution heat treated and aged at elevated temperatures.

This classification permits obvious generalizations on the response of each group to further elevated-temperature exposure.

SCOPE OF THE INVESTIGATION

This paper is concerned with the compressive properties of the following materials all tested in the form of 0.125-in. sheet:

Group A.... 24S-T
Group B.... 24S-T81, 24S-T86, 75S-T, and R301-T (clad)

With the exception of R301-T all were in the bare (not clad) condition. Specimens were stressed in the cross-grain direction. Testing temperatures included 150, 212, 250, and 300 F., with exposure periods ranging from ½ to 1000 hr. Room-temperature tests were performed to furnish a basis for comparison with the elevated-temperature results.

TABLE I.—ROOM TEMPERATURE TENSILE PROPERTIES OF THE MATERIALS TESTED.
(Average cross-grain values based on four tests for each material at 75 F.)

Material	Tensile Yield Strength (0.2 per cent offset), psi.	Ultimate Tensile Strength, psi.	Elongation in 2 in., per cent
24S-T (bare)...........	50 500	70 200	20.0
24S-T81 (bare)..........	65 900	70 400	7.0
24S-T86 (bare)..........	71 500	75 500	5.5
75S-T (bare)...........	75 400	83 700	11.0
R301-T (clad)..........	63 600	69 800	10.0

TABLE II.—NOMINAL COMPOSITIONS OF THE ALLOYS TESTED.

Material	Composition, per cent
24S............	4.5 copper, 0.6 manganese, 1.5 magnesium
R301...........	4.5 copper, 1.0 silicon, 0.8 manganese, 0.4 magnesium
75S............	The principal hardening elements are zinc and magnesium with smaller additions of copper and other elements. No further information is available.

NOTE.—In each case the remainder consists of aluminum and normal impurities.

TABLE III.—STANDARD HEAT TREATMENTS FOR THE MATERIALS TESTED.

Material	Heat Treatment
24S-T.........	24S alloy solution heat treated at 910 to 930 F. followed by quenching in cold water and aging at room temperature.
24S-T81........	24S-T stretched 1 per cent and aged 10 hr. at 375 F. or 12 hr. at 365 F.
24S-T86........	24S-T strain hardened by rolling to a reduction of about 5½ per cent and aged 5½ hr. at 375 F. or 8½ hr. at 365 F.
R301-T.........	R301 alloy solution heat treated at 930 to 950 F. followed by quenching in cold water and aging 6 hr. at 350 F. or 18 hr. at 320 F.
75S-T.........	75S alloy solution heat treated at 860 to 930 F. followed by quenching in cold water and aging 24 hr. at 250 F.

In the case of each material the following properties were determined:

1. Compressive yield strength (0.2 per cent offset),
2. Modulus of elasticity in compression (Young's modulus), and
3. Tangent modulus[3] for all stresses up to the yield strength.

Materials for specimens were obtained from the manufacturers' commercial stocks. The original room temperature tensile properties determined in tests on standard A.S.T.M. specimens (E 8 – 42),[4] are listed in Table I. The nominal chemical compositions of the alloys and the standard heat treatments for obtaining the various conditions are listed in Tables II and III.

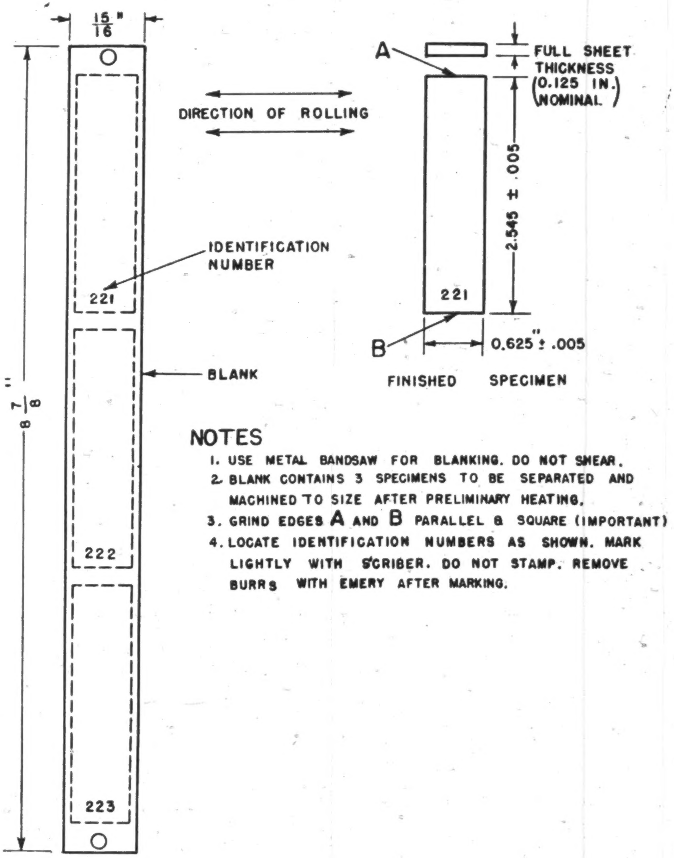
15"/16
8 7/8"
DIRECTION OF ROLLING
IDENTIFICATION NUMBER
221
222
223
BLANK
A
FULL SHEET THICKNESS (0.125 IN.) NOMINAL
2.545 ± .005
221
B
0.625" ± .005
FINISHED SPECIMEN
NOTES
1. USE METAL BANDSAW FOR BLANKING. DO NOT SHEAR.
2. BLANK CONTAINS 3 SPECIMENS TO BE SEPARATED AND MACHINED TO SIZE AFTER PRELIMINARY HEATING.
3. GRIND EDGES A AND B PARALLEL & SQUARE (IMPORTANT)
4. LOCATE IDENTIFICATION NUMBERS AS SHOWN. MARK LIGHTLY WITH SCRIBER. DO NOT STAMP. REMOVE BURRS WITH EMERY AFTER MARKING.

Fig. 1.—Compression Specimen and Blank Used in Elevated-Temperature Exposure.

Specimen Preparation and Preliminary Heating

All specimens of each material were obtained from a single sheet of 0.125-in. nominal thickness. Blanks were sawed to the dimensions shown in Fig. 1, following which they were heated for periods up to 1000 hr. in constant-temperature oil baths where they were held in fixtures to prevent warping. Temporal and spatial variations in the baths were no greater than ±2 F. After removal each blank was sawed into three individual specimens which were machined to the dimensions shown in Fig. 1. The bearing surfaces at the ends were finished by grinding. Specimens were then stored until needed, the interval between removal from the oil bath and subsequent testing ranging up to 30 days.

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[3] The tangent modulus is defined in a later section.

[4] Standard Methods of Tension Testing of Metallic Materials, 1944 Book of A.S.T.M. Standards, Part I, p. 962.

Before testing, the unmachined faces of each specimen were polished on a sheet of fine emery paper to remove small irregularities. Measurements of width and thickness were then made, the error in cross-section determination being less than $\frac{1}{2}$ per cent.

SPECIAL TESTING APPARATUS

Since in the past the compression testing of sheet materials at elevated temperatures has received little attention, the apparatus developed for the present tests will be described in some detail. It consists of three parts:

1. A fixture for supporting the specimen laterally and for insuring axial loading,
2. An oil-bath furnace to heat the fixture, to support it, and to transfer the load from the fixture to the lower platen of the testing machine, and
3. An averaging extensometer to be mounted on the specimen and to actuate a Tuckerman optical strain gage attached to extension arms outside of the furnace.

Testing Fixture:

Pack methods, roller guides, and lubricated plane guides have been used previously to prevent the buckling of sheet specimens tested in compression at room temperature. Hardened steel guides of the latter type are incorporated in the elevated-temperature fixture which is shown in Figs. 2, 3, and 5. The supporting surfaces are ground and polished to minimize friction. They are $\frac{1}{2}$ in. wide by $2\frac{7}{16}$ in. long, corresponding to specimen dimensions of 0.625 and 2.545 in., respectively. The clearances between specimen and guides may be controlled by means of two adjusting screws.

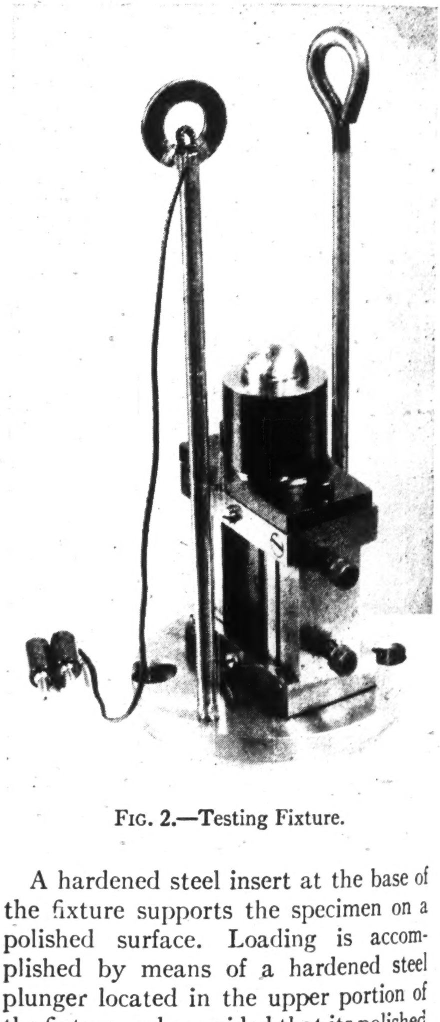

FIG. 2.—Testing Fixture.

A hardened steel insert at the base of the fixture supports the specimen on a polished surface. Loading is accomplished by means of a hardened steel plunger located in the upper portion of the fixture and so guided that its polished bearing surface is parallel to that of the insert. In testing, a hemispherical surface at the upper extremity of the plunger is contacted by the plane end of a loading ram attached to the cross-head of the testing machine. The contacting surfaces are hardened and ground.

Before insertion in the fixture, specimens were coated with a film of lubricant.[5] With this procedure no frictional difficulties were encountered at the temperatures employed.

Table IV, summarizing a series of tests on 0.125-in. 24S-RT sheet at room temperature, indicates that adjustment of the guide clearance screws is not critical so long as they are tightened by hand. Excessive values of yield strength characterize the tighter adjustments possible with a wrench. Throughout the testing program the screws were adjusted with moderate pressure by hand.

Since the differential expansion of specimen and fixture at elevated temperatures was small with respect to the allowable range of guide adjustments, no frictional difficulties arose from this source.

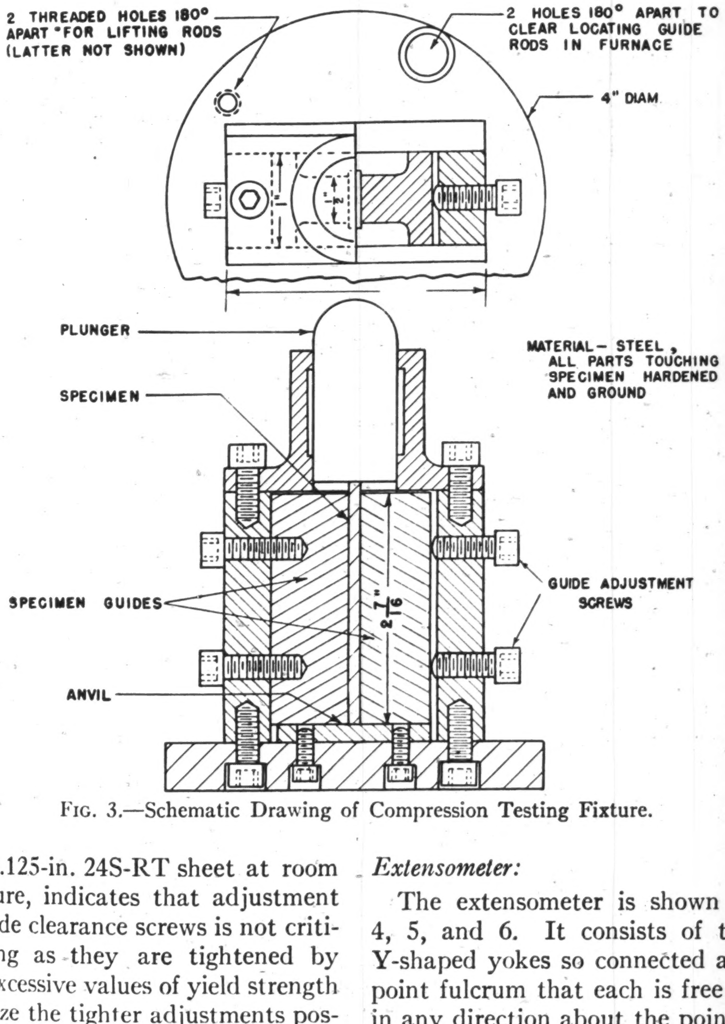
2 THREADED HOLES 180° APART FOR LIFTING RODS (LATTER NOT SHOWN)
2 HOLES 180° APART TO CLEAR LOCATING GUIDE RODS IN FURNACE
4" DIAM
1"
1/2"
PLUNGER
MATERIAL – STEEL, ALL PARTS TOUCHING SPECIMEN HARDENED AND GROUND
SPECIMEN
SPECIMEN GUIDES
2 7/16"
GUIDE ADJUSTMENT SCREWS
ANVIL

FIG. 3.—Schematic Drawing of Compression Testing Fixture.

Extensometer:

The extensometer is shown in Figs. 4, 5, and 6. It consists of two rigid Y-shaped yokes so connected at a midpoint fulcrum that each is free to pivot in any direction about the point of contact. The open end of each yoke is attached to the specimen by means of conically pointed screws. The opposite ends are designed to cause relative

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[5] Marfak No. 3, a wheel bearing lubricant previously employed in compression testing at the Bureau of Standards.

TABLE IV.—TESTS TO DETERMINE THE INFLUENCE OF GUIDE CLEARANCE.

Tests made at room temperature on ⅛-in. bare 24S-RT sheet stressed transversely.

Fixture No. 1, Extensometer No. 1, and Marfak No. 3 employed without use of oil bath.

Guide Adjustment	Test Number	Compressive Yield Strength, psi.	Modulus of Elasticity in Compression, psi.
Screws tightened loosely by hand	8-4-1	61 000	10 840 000
	8-4-9	60 900	10 720 000
	8-4-11	60 400	10 800 000
Average.		60 800	10 790 000
Screws tightened with moderate pressure by hand	8-4-2	60 900	10 950 000
	8-4-8	60 800	10 720 000
	8-4-10	60 600	10 790 000
Average.		60 800	10 820 000
Screws tightened as much as possible by hand	8-4-3	59 800	10 790 000
	8-4-7	60 900	10 650 000
	8-4-12	61 100	10 840 000
	8-4-14	61 600	10 880 000
Average.		60 900	10 790 000
Screws tightened with 4-in. wrench	8-4-4	64 200	10 770 000
	8-4-5	61 900	10 680 000
	8-4-6	64 200	10 760 000
	8-4-13	63 500	10 810 000
Average.		63 500	10 760 000

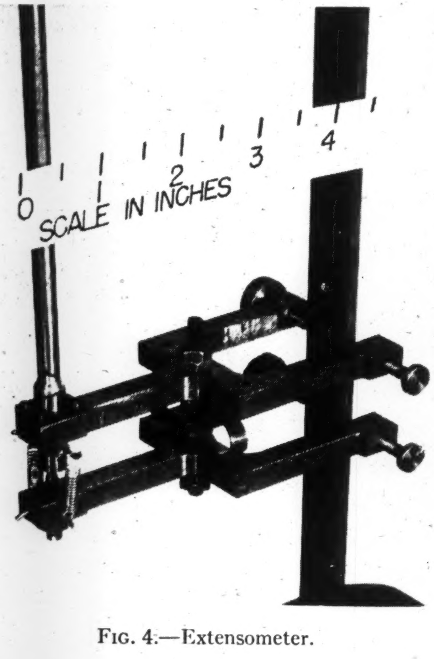

FIG. 4.—Extensometer.

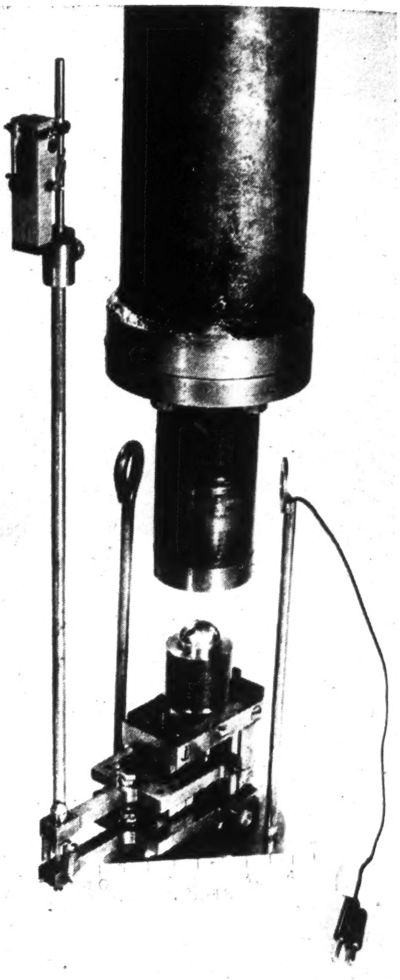

FIG. 5.—Fixture-Specimen-Extensometer Assembly.

longitudinal motion of a concentric rod and tube which in turn actuate a Tuckerman optical strain gage. With the extensometer mounted on a specimen in the oil bath, the Tuckerman gage may be observed readily in its location outside the furnace. The design of the yokes and fulcrum insures that a change in the Tuckerman reading is directly equivalent to an average value of the changes in length defined by sets of gage points located on opposite edges of the specimen. Thus the extensometer is direct reading and averaging. The fulcrum consists of a hardened 60-deg. cone held in a 90-deg. conical seat by means of a strong elliptical spring.

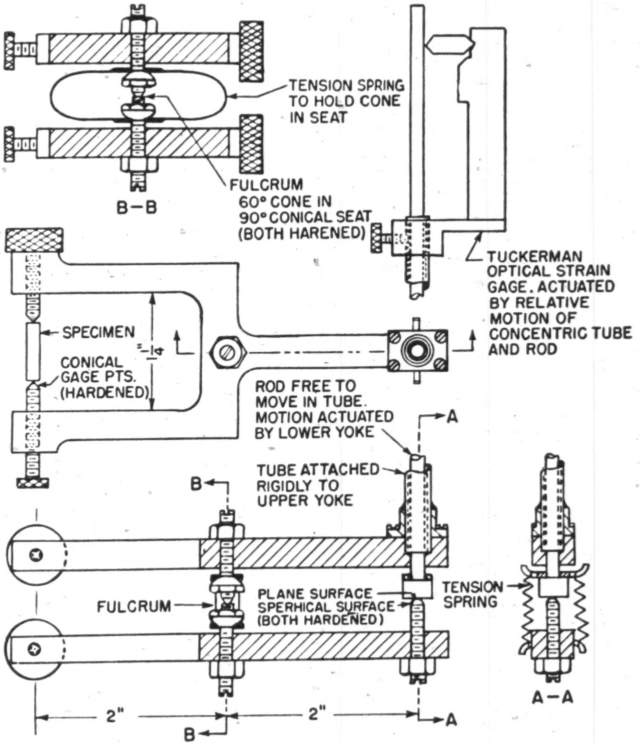
TENSION SPRING TO HOLD CONE IN SEAT
FULCRUM 60° CONE IN 90° CONICAL SEAT (BOTH HARENED)
B–B
TUCKERMAN OPTICAL STRAIN GAGE. ACTUATED BY RELATIVE MOTION OF CONCENTRIC TUBE AND ROD
SPECIMEN
CONICAL GAGE PTS. (HARDENED)
l
ROD FREE TO MOVE IN TUBE. MOTION ACTUATED BY LOWER YOKE
A
TUBE ATTACHED RIGIDLY TO UPPER YOKE
B
FULCRUM
PLANE SURFACE
SPERHICAL SURFACE
(BOTH HARDENED)
TENSION SPRING
A–A
2"
2"
B
A

FIG. 6.—Schematic Drawing of Extensometer.

Two fixtures and two extensometers were used alternately throughout the testing program, thus saving time and also allowing a continual check on the results since all tests were made in duplicate.

Preliminary tests summarized in Table V demonstrate the similarity of results obtained with duplicate sets of extensometers and fixtures. As in the case of Table IV, these tests were made on bare 24S-RT sheet at room temperature. The results of Tables IV and V are in good agreement with values obtained previously in investigations using other methods.[6]

Testing Furnace:

The electrically heated testing furnace is shown in Figs. 7 and 8. In addition to a thermostatically controlled oil bath, it contains a pedestal for support of the testing fixture. The surface of the pedestal is parallel to the base of the furnace but is thermally insulated from it by a layer of sheet mica. Centering of the fixture on the pedestal is insured by the use of vertical guide rods attached to the pedestal and passing through

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[6] R. L. Templin, E. C. Hartman, and D. A. Paul (3) of the Aluminum Research Laboratories cite the following values for 24S-RT sheet stressed transversely at room temperature:

Compressive yield strength.......	61 000 psi.
Modulus of elasticity in compression........................	10 700 000 psi.

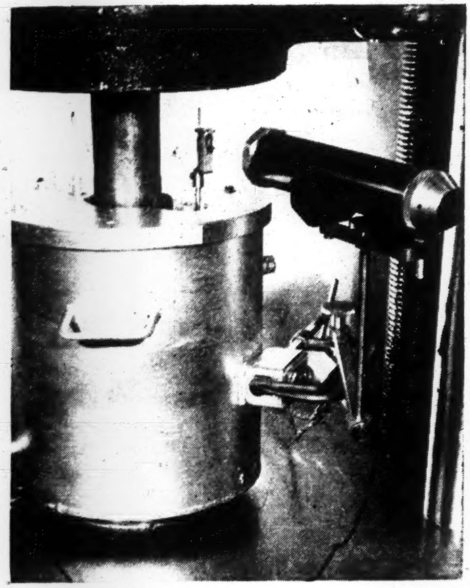


FIG. 7.—Test Assembly Showing Furnace, Loading Ram, Tuckerman Strain Gage, and Collimator.

TABLE V.—PRELIMINARY TESTS TO CHECK AGREEMENT OBTAINABLE WITH DUPLICATE FIXTURES AND EXTENSOMETERS.

All tests made at room temperature on ⅛-in. bare 24S-RT sheet stressed transversely. Guide clearance screws adjusted with moderate pressure by hand.

Test Number	Compressive Yield Strength, psi.	Modulus of Elasticity in Compression, psi.	Extensometer and Fixture	
A1	60 700	10 670 000	1	Tests made at room temperature without use of oil bath
A2	61 100	10 84[illegible] 000	1	
A3	61 300	10 620 000	1	
A4	60 900	10 780 000	1	
A5	61 800	10 560 000	1	
A6[a]	61 200	10 680 000	1	
A7[a]	61 400	10 630 000	1	
A8	61 100	10 880 000	1	
Average	61 200	10 710 000		
B3	60 300	10 880 000	2	Tests made at room temperature without use of oil bath
B4	61 200	10 640 000	2	
B5	61 100	10 870 000	2	
B6	60 800	10 770 000	2	
B7	60 900	10 710 000	2	
B8	60 700	10 890 000	2	
B9	61 000	10 700 000	2	
B10	60 900	10 630 000	2	
Average	60 900	10 760 000		
B11	60 700	10 700 000	1	Tests made in oil bath at room temperature
B12	61 100	10 730 000	2	
B13	61 400	10 640 000	1	
Average	61 100	10 690 000		

[a] Two tests made without lubricant (guides wiped clean) to test sensitivity to changes in lubrication. All other tests made as usual with Marfak No. 3.

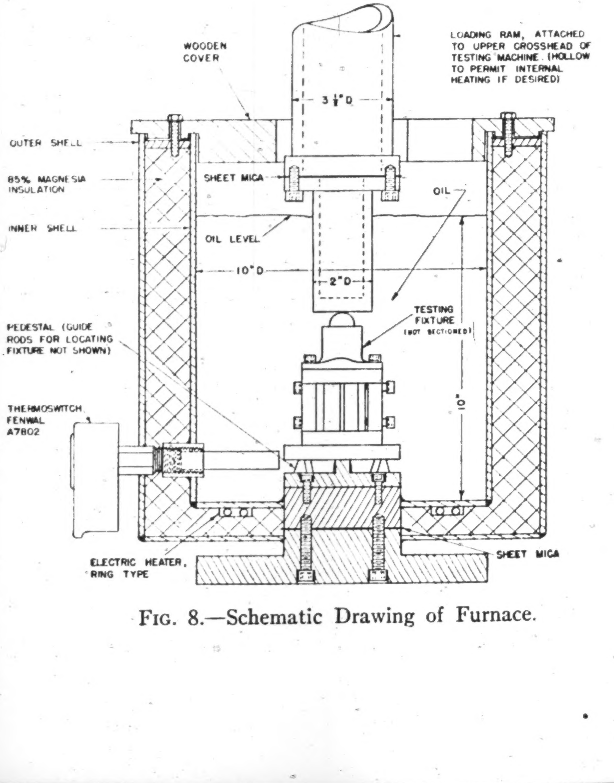


FIG. 8.—Schematic Drawing of Furnace.

matching holes in the base of the fixture. The bearing surface of the pedestal consists of a number of high, narrow cleats so arranged that debris may not interfere with the seating of the fixture. The fixture may be inserted and removed through an opening in the top of the furnace, the latter also serving to admit the loading ram. As in the case of the pedestal, the ram contains an insulating layer of mica to minimize heat loss by conduction.

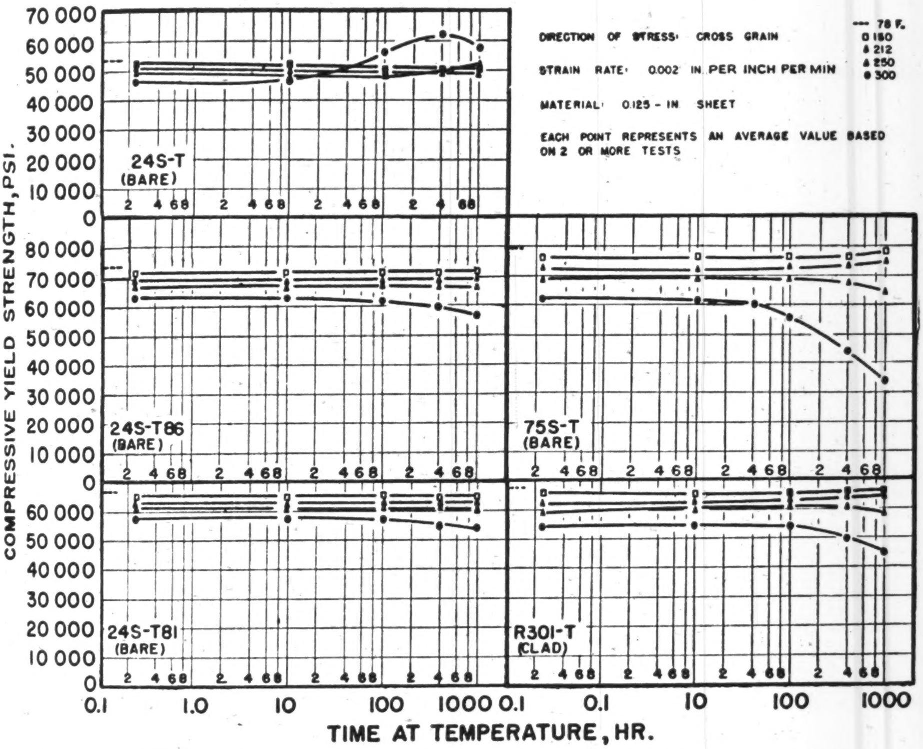


FIG. 9.—Effects of Temperature and Exposure Time on Compressive Yield Strength at Elevated Temperatures.

A "Core Oven Conveyor Oil" was employed in the oil bath at all temperatures. No fuming was noted and forced circulation was not required. Under routine testing conditions at 300 F., the over-all temperature variation over the length of a specimen was less than 1 F. after attainment of equilibrium. In each test the specimen temperature was measured by means of a thermocouple located in the base of the fixture, the temperature at this location corresponding precisely to that of the specimen. All testing temperatures were within ±2 F. of the nominal value.

It had been planned to heat the hollow ram internally to compensate for heat lost to the atmosphere during its brief withdrawal from the furnace between tests. It was found, however, that such heating was unnecessary, the loss being small in relation to the heat capacity of the oil bath.

A second furnace similar to the first

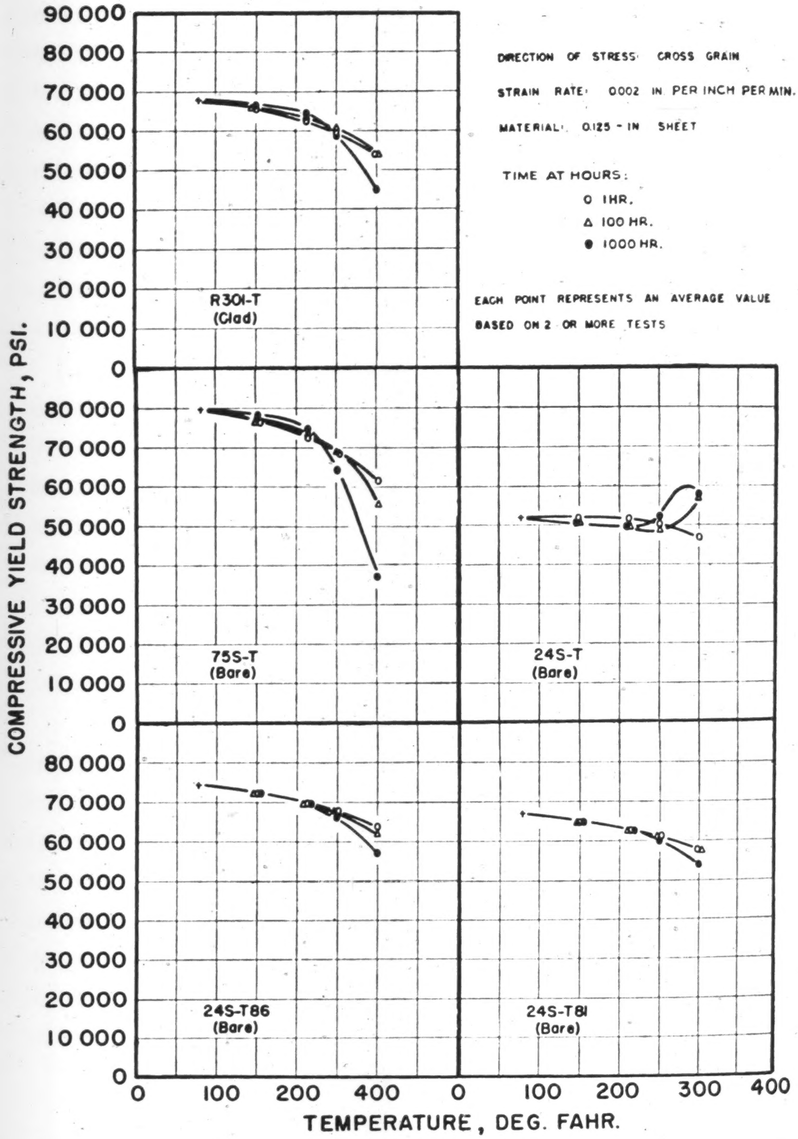


FIG. 10.—Effects of Temperature and Exposure Time on Compressive Yield Strength at Elevated Temperatures.

was used to preheat specimens just prior to insertion in the testing furnace.

TESTING PROCEDURE

Preceding each test, the specimen faces were polished and measurements of thickness and width were obtained. The specimen was then coated with lubricant and inserted in the fixture. After attachment of the extensometer, the entire assembly was placed in the oil bath of the preheating furnace where it was allowed to remain for approximately ten minutes. During this period it attained a temperature within several degrees of the desired testing temperature. At this stage the ram was raised from the testing heating bath. The specimen was loaded continuously throughout the test. Following removal of the apparatus from the testing furnace it was washed in a bath of solvent at room temperature and

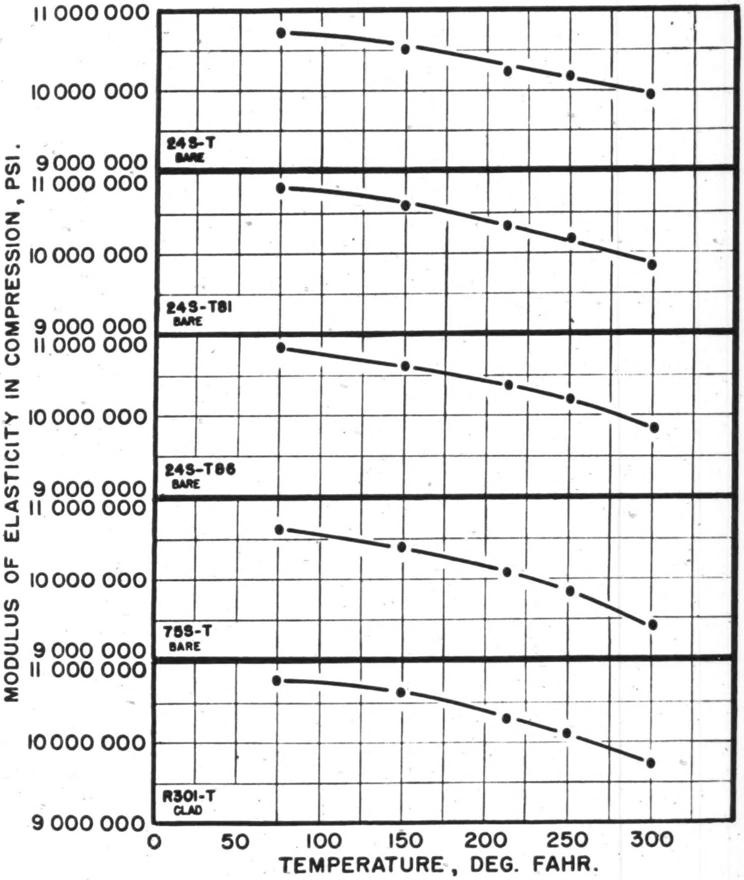
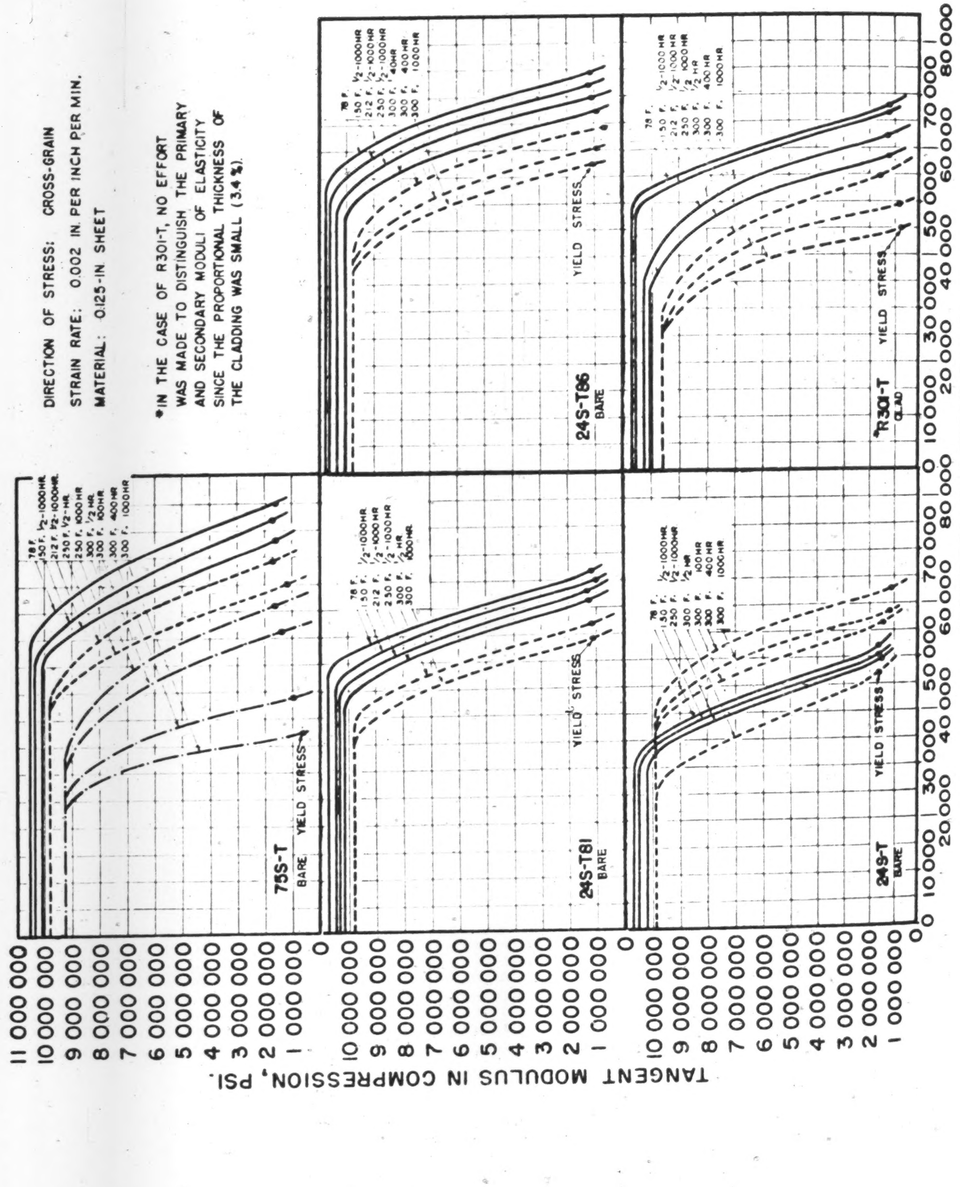


Fig. 11.—Effects of Temperature on the Modulus of Elasticity in Compression at Elevated Temperatures.

furnace, the fixture was transferred from the preheating bath, and the ram was replaced. The Tuckerman strain gage was then attached and the entire assembly allowed to attain thermal equilibrium. Testing at a constant strain rate of 0.002 in. per inch per minute was begun approximately 20 min. after insertion of the assembly in the preheating furnace, another specimen was inserted in the fixture.

All tests were conducted on the 10,000-lb. range of a 200,000-lb. Tate-Emery testing machine. Use of the preheating furnace and duplicate fixtures and extensometers permitted the simultaneous progress of two tests, thus reducing the time required for testing.


DIRECTION OF STRESS: CROSS-GRAIN
STRAIN RATE: 0.002 IN. PER INCH PER MIN.
MATERIAL: 0.125-IN SHEET
*IN THE CASE OF R301-T, NO EFFORT WAS MADE TO DISTINGUISH THE PRIMARY AND SECONDARY MODULI OF ELASTICITY SINCE THE PROPORTIONAL THICKNESS OF THE CLADDING WAS SMALL (3.4%).
75S-T BARE
78 F. 1/2-1000HR.
150 F. 1/2-1000HR.
212 F. 1/2-1000HR.
250 F. 1/2-HR.
250 F. 1000HR.
300 F. 1/2HR.
300 F. 100HR.
300 F. 400HR.
300 F. 1000HR.
24S-T86 BARE
78 F. 1/2-1000HR
150 F. 1/2-1000HR
212 F. 1/2-1000HR
250 F. 1/2-1000HR
300 F. 1/2HR
300 F. 400HR
300 F. 1000HR
24S-T81 BARE
78 F.
150 F. 1/2-1000HR
212 F. 1/2-1000HR
250 F. 1/2-1000HR
300 F. 1/2HR
300 F. 1000HR
R301-T* CLAD
78 F.
150 F. 1/2-1000HR
212 F. 1/2-1000HR
250 F. 1/2 1000HR
300 F. 1/2HR
300 F. 400HR
300 F. 1000HR
24S-T BARE
78 F.
150 F. 1/2-1000HR
250 F. 1/2-1000HR
300 F. 1/2HR
300 F. 100HR
300 F. 400HR
300 F. 1000HR
YIELD STRESS
TANGENT MODULUS IN COMPRESSION, PSI.
11 000 000
10 000 000
9 000 000
8 000 000
7 000 000
6 000 000
5 000 000
4 000 000
3 000 000
2 000 000
1 000 000
0
COMPRESSIVE STRENGTH, PSI.
0
10000
20000
30000
40000
50000
60000
70000
80000

DERIVATION OF TANGENT MODULUS CURVES

When the design must take plastic buckling into consideration it is desirable to know the shape of the stress-strain curve in the plastic region, the relation between stress and the slope of the curve being a significant feature (**4, 5**). The slope is often called the "tangent modulus" and the curve representing it as a function of stress is known as the "tangent modulus curve." The latter is essentially equivalent to the stress-strain curve itself, since either type may be constructed directly from the other.

The tangent modulus curves of this paper were constructed in the following manner: The stress-strain curve was determined for each test and a series of tangents was drawn at convenient stress levels. The slopes of the tangents were then measured and plotted against the corresponding stresses. Smooth curves were faired through the points obtained in this way.

EXPERIMENTAL RESULTS

Test results are shown by means of curves in Figs. 9 to 12. Figures 9 and 10 describe the effects of temperature and exposure time on compressive yield strength (0.2 per cent offset), each point representing an average value based upon two or more tests. Figure 10 is a crossplot of Fig. 9.

Figure 11 shows the effect of temperature on the modulus of elasticity. This property appears to be independent of the time at temperature. Each point represents an average of at least ten tests.

Figure 12 shows the variation of the tangent modulus with stress, temperature, and time. At temperatures where the effect of time is small, single curves have been drawn to represent all times from ½ to 1000 hr.

Effects of Temperature and Time on Compressive Yield Strength:

The effects of elevated-temperature exposure on compressive yield strength (Figs. 9 and 10) are mainly of two types:

1. Changes in yield strength produced by the effect of temperature alone, independent of precipitation effects, and
2. Changes in yield strength resulting from structural alteration of the material, dependent upon time as well as temperature.

In the case of the shortest exposure time (½ hr.) the principal effect is that of temperature alone, increases in temperature producing lower values of yield strength.

As would be expected on the basis of previous heat treatment, the response of 24S-T to continued exposure is found to differ from that of the other materials. All are amenable to artificial aging and in the process of production all except 24S-T have been aged at elevated temperatures to produce conditions approximating the maximum attainable yield strength. Except in the case of 24S-T, therefore, further exposure may be expected to result in "overaging" attended by a decrease in yield strength. In the case of 24S-T an initial increase is to be expected followed in time by attainment of a maximum and thereby overaging. The results noted in Fig. 9 are generally in accord with these expectations.

For periods up to 1000 hr. at temperatures as high as 250 F. the effect of exposure time is small, an indication that at such temperatures the anticipated changes are sluggish. An exception is noted in the case of 75S-T where an appreciable decrease in yield strength is apparent after 1000 hr. at 250 F. At 300 F. all materials exhibit changes after 1000 hr. and again the effect is most marked in the case of 75S-T where the strength is 45 per cent lower than the ½-hr. value. At this temperature 24S-T reaches a maximum value in approximately 400 hr.

The tendency of 75S-T to undergo loss of strength more rapidly than the other materials is consistent with its lower temperature for artificial aging (Table III).

It may be noted in Fig. 9 that with continued exposure at the lower temperatures small increases in yield strength occur in R301-T and 75S-T while small decreases are found in 24S-T. The increases in R301-T and 75S-T suggest an originally underaged condition in these materials. The decrease in the latter case is consistent with the small decreases known to occur in the preliminary stages of the artificial aging of 24S-T (1).

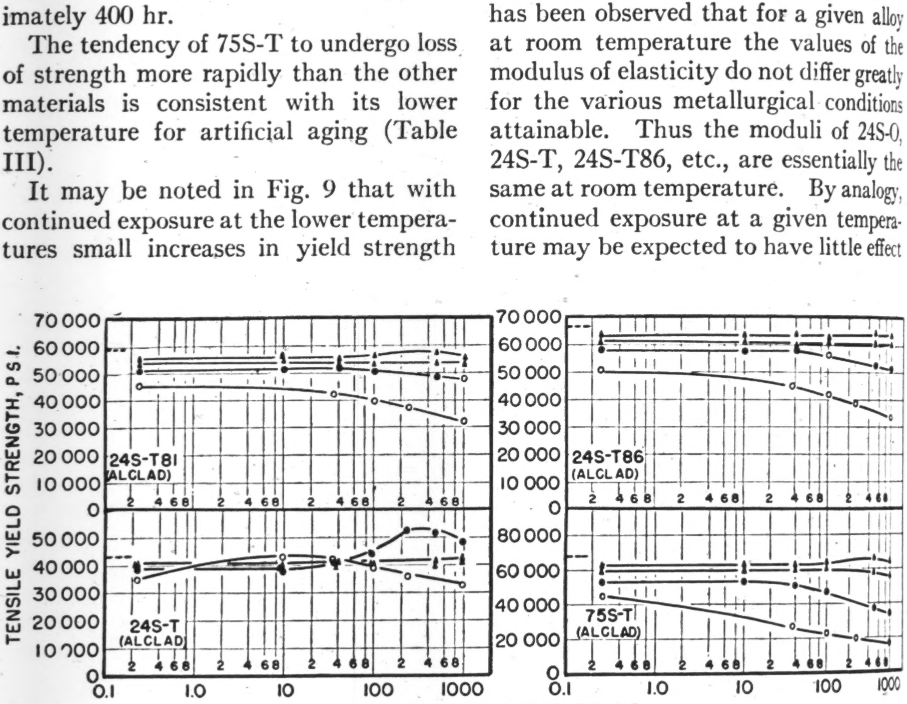


FIG. 13.—Effects of Temperature and Exposure Time on Tensile Yield Strength at Elevated Temperatures.

Effects of Temperature and Time on the Modulus of Elasticity:

As anticipated, values of the modulus of elasticity are found to decrease with increasing temperature. This property has been plotted as a function of temperature alone in Fig. 11 since the original data indicate it to be independent of exposure time. The latter feature, which was noted also in elevated temperature tension tests (6), is not unexpected. It has been observed that for a given alloy at room temperature the values of the modulus of elasticity do not differ greatly for the various metallurgical conditions attainable. Thus the moduli of 24S-O, 24S-T, 24S-T86, etc., are essentially the same at room temperature. By analogy, continued exposure at a given temperature may be expected to have little effect on the modulus in spite of the fact that extensive metallurgical changes may occur during exposure.

Effects of Temperature and Time on the Tangent Modulus:

The effects of temperature, time, and stress on the tangent modulus are shown for all testing conditions in Fig. 12. The relatively minor effect of exposure time at the lower temperatures has been neglected and single curves have been drawn to represent all times from ½ to 1000 hr.

As noted previously, the initial portion of each curve (the modulus of elasticity) is determined by temperature alone. At temperatures where time is an important factor its influence is evident at the proportional limit and at higher stresses.

Since R301-T is a clad material it should exhibit both primary and secondary moduli of elasticity. In the 0.125-in. sheet used in this investigation, however, the proportionate thickness of the cladding was small ($3\frac{1}{2}$ per cent) and the effect was scarcely detectable. Accordingly, no effort was made to distinguish two moduli in drawing stress-strain curves. Instead a single straight line was found to fit the data satisfactorily up to the proportional limit.

Comparison of Compressive and Tensile Yield Strengths:

Comparison of the compressive data with tensile results obtained previously (6) indicates that the values of tensile and compressive yield strength respond in similar manner to elevated-temperature exposure. (Compare Fig. 9 with Fig. 13 which has been reproduced from reference 6.) For given conditions, the compressive values are greater than the tensile values due in part to the use of clad material in the tension tests. It may be seen, however, that the compression data may be approximated closely by calculations based on the tension data and on knowledge of the compressive properties at room temperature.

Let us suppose, for instance, that it is desired to calculate the compressive yield strength of bare 0.125-in. 75S-T sheet at 300 F. after an exposure of 100 hr. According to Fig. 13, the tensile value for this condition is 46,000 psi. (for 0.040-in. clad sheet). The tensile yield strength of the clad material at room temperature is 67,000 psi. (Fig. 13) while the correspondent compressive value for bare 0.125-in. sheet is 79,400 psi. (Fig. 9). We may then calculate the desired value as

$$\frac{79{,}400}{67{,}000} \times 46{,}000 = 54{,}500 \text{ psi.}$$

From Fig. 9 the actual yield strength is seen to be 55,500 psi., in fair agreement with the calculated value. Table VI has been derived in a similar manner from Fig. 13 to illustrate the agreement obtained over a wide range of conditions.

TABLE VI.—EXPERIMENTAL VALUES OF COMPRESSIVE YIELD STRENGTH AT ELEVATED TEMPERATURES COMPARED WITH VALUES CALCULATED FROM TENSION DATA.

Treatment	Compressive Yield Strength, psi. $\times 10^{-3}$							
	24S-T		24S-T81		24S-T86		75S-T	
	Experimental[a]	Calculated[b]	Experimental[a]	Calculated[b]	Experimental[a]	Calculated[b]	Experimental[a]	Calculated[b]
77 F.	51.9	51.9	66.8	66.8	74.3	74.3	79.4	79.4
212 F., ½ hr.	51.0	49.4	62.8	63.3	70.0	70.9	72.5	75.8
212 F., 100 hr.	49.5	49.4	62.8	63.3	70.0	70.9	74.0	75.8
212 F., 1000 hr.	49.5	50.6	62.8	63.3	70.0	70.9	74.5	75.8
250 F., ½ hr.	48.8	48.2	61.0	61.0	67.5	68.1	69.0	71.1
250 F., 100 hr.	48.5	48.2	61.0	61.0	67.5	68.1	69.0	71.1
250 F., 1000 hr.	52.0	50.6	61.0	61.0	66.2	67.0	64.2	64.0
300 F., ½ hr.	46.5	47.0	57.6	57.6	63.7	64.8	61.5	61.6
300 F., 100 hr.	56.5	54.2	57.0	57.1	62.2	62.6	55.5	54.5
300 F., 1000 hr.	58.0	59.0	53.7	53.1	57.0	57.0	37.5	39.1

[a] From Fig. 9 for 0.125-in. bare sheet.

[b] From the relation

$$CYS = TYS \frac{(CYS)_{RT}}{(TYS)_{RT}}$$

where:

CYS = compressive yield strength of 0.125-in. bare sheet for any temperature and time, cross-grain;

TYS = tensile yield strength of 0.040-in. clad sheet for corresponding conditions (from Fig. 13);

$(CYS)_{RT}$ = compressive yield strength of 0.125-in. bare sheet at room temperature (from Fig. 9); and

$(TYS)_{RT}$ = tensile yield strength of 0.040-in. clad sheet at room temperature (from Fig. 13). The values used above are 43,100, 59,100, 66,500, and 67,000 psi. for 24S-T, 24S-T81, 24S-T86, and 75S-T, respectively.

The method appears to correct for the presence or absence of cladding, since the compression values for bare sheet have been derived from the tensile values of the clad material. Both sets of data apply to the cross-grain direction. The method would probably prove less successful if applied to the rolling direction.[7] It is certainly not suggested that it is generally applicable for all materials and conditions.

The Problem of Creep:

As in the case of tension testing, the results of short-time compression tests at elevated temperatures may be influenced to a considerable degree by the strain rate employed. At sufficiently high temperatures extensive plastic deformation may occur under prolonged loading at stresses well below the value of the short-time "yield strength." Under the same conditions, the tangent modulus data may also lose significance. Such data, therefore, should be used with caution.

Figure 14 is presented to indicate the influence of creep at 300 F. The data were obtained for a 24S-T81 specimen loaded in the usual manner and maintained for 30 min. at a constant compressive stress slightly lower than the yield strength. It may be seen that considerable deformation occurred after attainment of this stress.

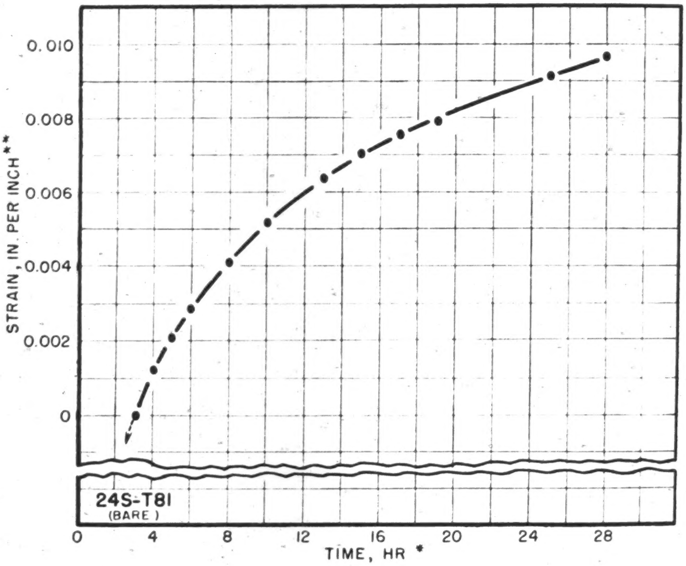


FIG. 14.—Creep of 24S-T81 Specimen at 300 F. under Constant Compressive Stress Approximating the Short-Time Compressive "Yield Strength."

* Time measured from beginning of test.
** Strain measured as difference between instantaneous value and strain immediately after stress was applied.

Choice of Materials for Elevated Temperature Service:

On the basis of the preceding data it is possible to rank the high-strength aluminum alloys with regard to suitability for services at elevated temperatures. The rating is intended to apply, of course, only with respect to the compressive properties. Of the materials tested, members of the 24S-T8 series appear to offer the best possibilities. The behavior of R301-T is similar to that of 24S-T81 but its loss in yield strength is more rapid at 300 F. After sufficiently long exposure 24S-T may approach or exceed the strength of these materials but until fully aged it is lower in strength and may be more susceptible to corrosion.[8] At the lower temperatures 75S-T exhibits superior properties but its loss in yield strength is extremely rapid at 300 F.

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[7] According to references 3 and 7 the cross-grain values of yield strength for several aluminum alloy sheet materials at room temperature are nearly the same in tension as in compression. In the rolling direction, however, the values are considerably greater in tension than in compression. It is possible that the relative excess in the latter case might be altered at higher temperatures.

Summary and Conclusions

The tests to determine the short-time compressive properties of five high-strength aluminum alloy sheet materials at temperatures up to 300 F. after exposure times ranging from $\frac{1}{2}$ to 1000 hr., indicated that the effects of temperature and time on the compressive yield strength and modulus of elasticity are similar to their effects on the corresponding tensile properties. For the materials tested it is found that approximate values of compressive yield strength may be derived by calculations based upon the elevated-temperature properties in tension.

Of the materials tested it is found that 24S-T86 and 24S-T81 offer the best possibilities for elevated-temperature service (judged solely on the basis of compressive properties). The behavior of R301-T is similar to that of 24S-T81 but its loss of strength is more rapid at 300 F. After sufficiently long exposure 24S-T may approach or exceed the strength of those materials but until fully aged it is lower in strength and may be more susceptible to corrosion. At the lower temperatures 75S-T exhibits superior properties but its loss in strength is rapid at 300 F.

Acknowledgment:

The authors are grateful for sponsorship by the War Production Board and for supervision by the Office of Production Research and Development. They wish also to acknowledge the extensive contributions made by Frank Brezee, Philip Carrera, and Woodrow F. Harper, NRC-548 engineers who participated in the testing program.

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(2) T. L. Fritzlen and L. F. Mondolfo, "Heat Treatment of R301 Alloy," *Metals Progress*, Vol. 47, No. 6, June, 1945, pp. 1128–1136.

(3) R. L. Templin, E. C. Hartmann, and D. A. Paul, "Typical Tensile and Compressive Stress-Strain Curves for Aluminum Alloy 24S-T, Alclad 24S-T, 24S-RT, and Alclad 24S-RT Products," *Technical Paper No. 6*, Aluminum Research Laboratories (1942).

(4) W. L. Howland, "Variation of the Modulus of Elasticity of Duralumin," *Journal Aeronautical Sci.*, Vol. 4, No. 12, October, 1937, pp. 507–509.

(5) J. R. Fishel, "The Compressive Strength of Thin Aluminum Sheet in the Plastic Region," *Journal Aeronautical Sci.*, Vol. 8, No. 10, August, 1941, pp. 373–383.

(6) A. E. Flanigan, L. F. Tedsen, and J. E. Dorn, "Tensile Properties of Aluminum Alloy Sheet at Elevated Temperatures," Tech. Pub. No. 1929, Am. Inst. Mining and Metallurgical Engrs., December, 1945.

(7) "ANC-5, Strength of Aircraft Elements," Army-Navy Civil Committee on Aircraft Design Criteria, December, 1942.

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[8] In the case of 24S it has been found that there is an increase in susceptibility to corrosion during the first stages of elevated-temperature aging. With continued aging, however, corrosion resistance is restored until in the fully aged condition it approximates that resulting from natural aging at room temperature (1).

DISCUSSION

Mr. F. R. Shanley[1] (*presented in written form*).—As a member of the committee which originally requested that this work be done for the aircraft industry, I might explain why we wished to have the data in the temperature range and in the form given by the authors. The need for data up to 300 F. was precipitated by the use of heat de-icing, in which the structure is heated by exhaust gases or other means. The tangent-modulus curves (Fig. 12) were requested because we use such data a great deal in predicting the buckling strength of aircraft structures.

To illustrate the usefulness of the tangent-modulus curves shown in Fig. 12, I have selected four of them and converted the data into column curves, as shown in the accompanying Fig. 15. The Engesser column formula was used. This is nothing more than the classical Euler formula in which the modulus of elasticity, E, is replaced by the tangent modulus, E_t. The calculations can be made in a few minutes by selecting a number of values of stress, picking the corresponding values of E_t from the curves of Fig. 12, and substituting both in the Engesser equation to find the corresponding value of slenderness ratio (L/ρ).

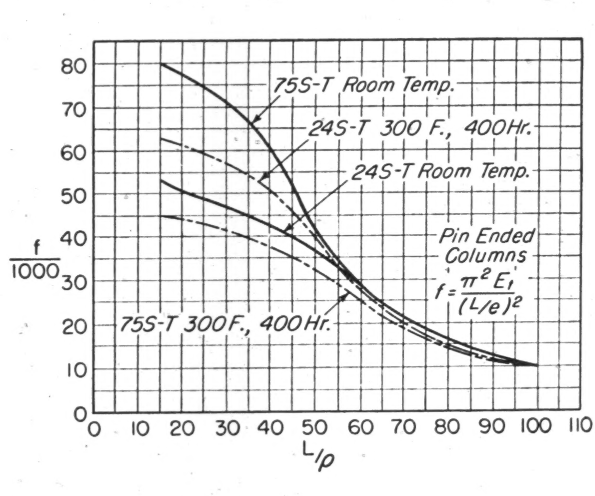

Fig 15.—Column Curves Based on Tangent Modulus, at Different Temperatures.

The materials selected from their Fig. 12 were 24S-T and 75S-T, at room temperature and at 300 F. held for 400 hr. The superiority of 75S-T at room temperature is very obvious for the shorter columns. At a slenderness ratio of 30, the 75S-T column develops a buckling stress about 50 per cent higher than that for 24S-T. At 300 F. the buckling stress for the 24S-T column is increased, while that for 75S-T is greatly decreased. At a slenderness ratio of

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[1] Chief of Engineering Research, Lockheed Aircraft Corp., Burbank, Calif.

30, the 75S-T column loses over 40 per cent of its room temperature strength, while the 24S-T column is about 20 per cent stronger than it was at room temperature. Although these curves were plotted for primary (bending) failure of simple columns, they also indicate what might be expected for any form of buckling.

The use of the tangent modulus in strength calculations is rapidly being extended in aircraft work. It eliminates arguments about the significance or definition of the yield strength and it takes care of the shape of the stress-strain diagram at the "knee." The present paper shows how this method can account for temperature effects. Further information on the use of the tangent modulus can be found in "Non-Dimensional Buckling Curves—Their Development and Application" by Cozzone and Melcon.[2]

MR. JOHN E. DORN (*author's closure by letter*).—The authors appreciate Mr. Shanley's pertinent discussion on the application of the tangent moduli data presented in this paper to the design of columns for resistance to buckling. The illustrations given in his contribution to this work reveal the need for these data for accurate design of thermal anti-icing equipment.

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[2] Paper presented at the Fourteenth Annual Meeting of the Institute of the Aeronautical Sciences, January, 1946, New York, N. Y.

TENSILE AND CREEP STRENGTHS OF SOME MAGNESIUM-BASE ALLOYS AT ELEVATED TEMPERATURE[*]

BY A. A. MOORE[1] AND J. C. MCDONALD[1]

SYNOPSIS

Creep tests up to and including rupture were carried out for periods of 1000 hr. Several commercial alloys, as well as some experimental cerium-containing alloys, were studied.

At room temperature it was observed for many of the alloys that the yield strength was an adequate criterion for design of parts to be loaded steadily for long periods. At the maximum temperature of test, 300 F., this is true of cerium-containing alloys, but the commercial alloys have strengths-to-rupture less than the yield strength. The magnesium-manganese alloy, M, is the best of the commercial alloys in respect to creep properties at elevated temperature.

Magnesium-base alloys do not lose ductility at rupture under long-time conditions of loading.

The application of materials in the manufacture of heat engines has led to many studies of the properties of the materials at the operating temperatures of the engines. The subject has assumed increasing importance as ever higher stresses are encountered in going to the light-weight engines required by modern aircraft. Margins of safety become smaller, making the designer more conscious of the properties he may expect under actual running conditions. In developing better materials, the first basis of comparison with the familiar materials is the strength of a test bar.

The results of the standard static tension test have been widely used. Of late years it has become increasingly recognized that such data are insufficient and may be misleading. Over a long time, creep can occur under steady stresses. Deformation may be so extensive as to ruin the part for any further use, or actual rupture may occur. For these reasons, the effect of steady stresses over a long period of time must be known. The fatigue strength is also of importance. Such data for magnesium alloys may be found elsewhere.[2]

Magnesium alloys are already widely used in aircraft engines. This paper presents data on commercial alloys which are relatively stable at operating temperatures of such engines. Some properties are also presented on magnesium-base alloys containing cerium. Similar alloys have been used on German engines. They retain their creep strength to higher temperatures than present commercial American alloys.[3]

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[*] Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] The Dow Chemical Co., Midland, Mich.

[2] G. H. Found, "The Notch Sensitivity in Fatigue Loading of Some Magnesium-Base and Aluminum-Base Alloys," *Preprint No. 35*, Forty-Ninth Annual Meeting, Am. Soc. Testing Mats., June 24–28, 1946.

[3] T. E. Leontis and J. P. Murphy, "The Properties of Cerium Containing Magnesium Alloys at Room and Elevated Temperatures," T. P. 1995, *Metals Technology*, April, 1946.

A time of 1000 hr. has been arbitrarily selected as the limit for the creep studies. Shorter times, 100 hr., have been used in preliminary work and in alloy evaluation. Some applications would probably require times of test longer than 1000 hr., but in order to obtain data on several materials, it appeared desirable to limit the test to 1000 hr. because of the finite testing capacity available.

TABLE I.—MATERIALS TESTED.

Designation		Nominal Composition, per cent				Typical Properties at Room Temperature		
A.S.T.M.	Dow	Aluminum	Cerium	Manganese	Zinc	Tensile Strength, psi.	Tensile Yield Strength, psi.	Elongation, per cent
				Castings				
AZ92	C-S[a]	9	0	0.2	2	24 000	14 000	2
AZ92	C-HTA[b]	9		0.2	2	40 000	23 000	2
AZ92	C-HTS[c]	9		0.2	2	40 000	20 000	3
AZ63	H-S[a]	6		0.2	3	29 000	14 000	5
AZ63	H-HTA[b]	6		0.2	3	40 000	19 000	5
AZ63	H-HTS[c]	6		0.2	3	40 000	17 000	7
AZ90	R[d]	9		0.2	0.7	33 000	22 000	3
E10	....	....	10	....	....	....	....	....
				Forgings				
M1	M	....	....	1.5	....	36 000	23 000	7
AZ80	O-1 HTA[b]	8.5	....	0.2	0.5	50 000	34 000	5
EM22	....	....	2	2	....	....	....	....
				Extrusions				
M1	M	....	....	1.5	....	38 000	26 000	10
AZ31	FS-1	3	....	0.2	1	40 000	30 000	15
AZ61	J-1	6	....	0.2	1	40 000	32 000	15
AZ80	O-1 HTA[b]	8.5	....	0.2	0.5	50 000	34 000	7
				Sheet				
M1	Ma	....	....	1.5	....	33 000	15 000	17
M1	Mh	....	....	1.5	....	37 000	29 000	8

[a] Sand-cast and stabilized 4 hr. at 500 F.
[b] Heat-treated and aged 16 hr. at 350 F.
[c] Heated-treated and stabilized 4 hr. at 500 F.
[d] Die cast.

MATERIALS TESTED

Table I lists the materials tested. Composition, designation, and typical tensile properties at room temperature are given. Generally speaking, these materials are relatively stable up to at least 300 F. Tension test results at temperatures to 600 F. are reported later in the paper, although most of the materials could not be recommended for use at that temperature.

The chief omissions of commercial alloys from this paper are solution heat-treated castings and hard-rolled sheet. These materials are unstable above 300 F., and are ordinarily not recommended for use at elevated temperatures.

The amount of data presented varies among the alloys. This was due to concentration of the test program on items which seemed of greatest importance at the time.

METHODS OF TESTING

Three types of tests were made: short-time tension tests and two forms of creep tests differing mainly in the accuracy of strain measurements. At high

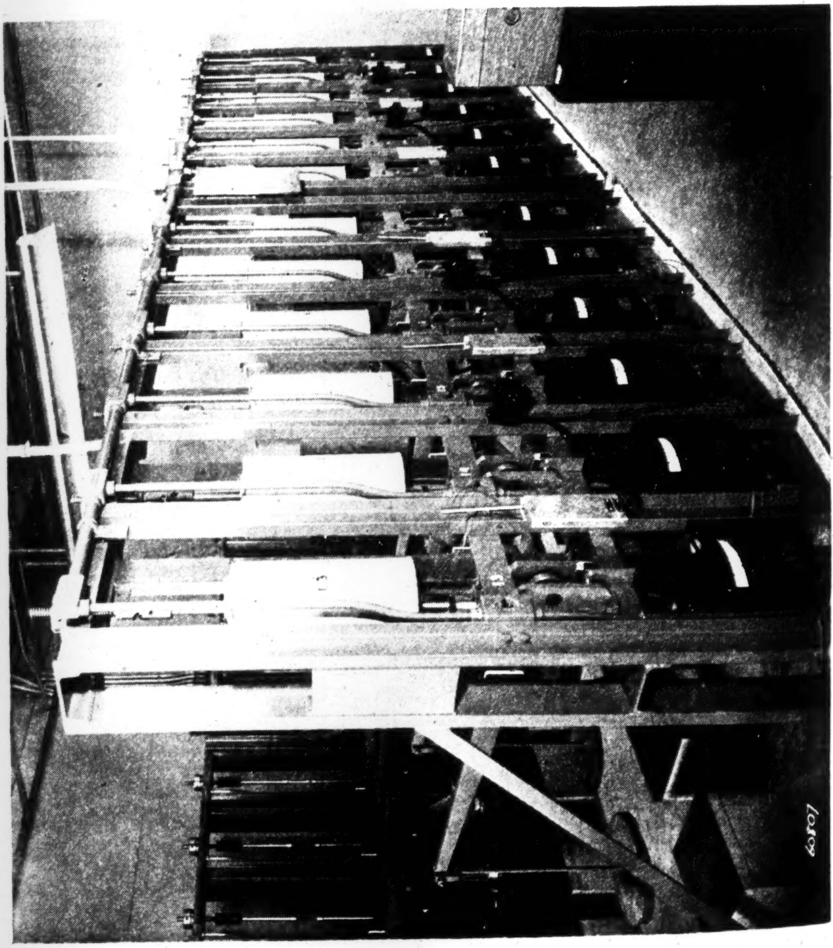

FIG. 1.—Creep-to-Rupture Tests at Room and Elevated Temperatures.

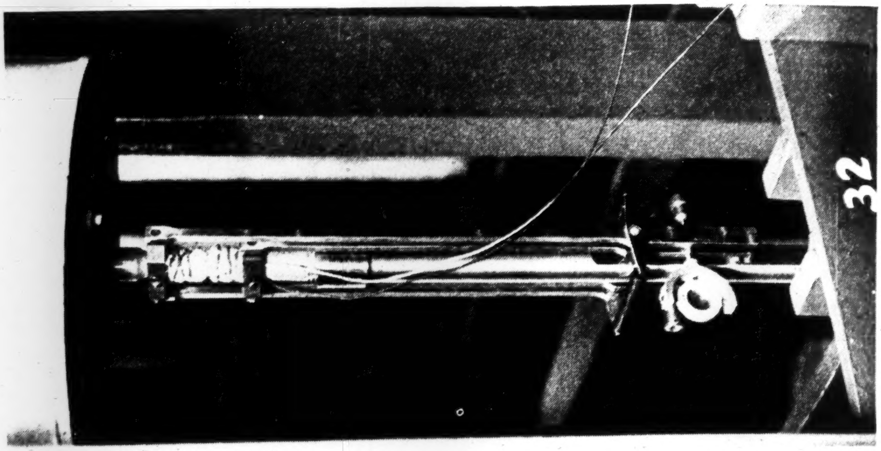

FIG. 2.—Extensometer System for Elevated Temperature Tests.

stress levels, the specimen ruptures and a less sensitive measurement is required.

The short-time high-temperature tension tests were made in accordance with the procedures given in Standard Recommended Practice for Short-Time Elevated-Temperature Tension Tests of Metallic Materials (E 21 – 43)[4] with the exception of specimen size and time at temperature. On some of the material tested, round bars 0.505 in. in diameter could not be obtained, so standard sheet tension test specimens, as given in Standard Methods of Tension Testing of Metallic Materials (E 8 – 42)[5] were used. The round test specimens had threaded shoulders which were fitted into shackles supported through spherical seats at the cross-heads. The flat specimens were pin-connected to clevis type adapters which in turn were threaded to fit the same shackles.

The time at temperature was reduced to 10 min. instead of the 1 hr. recommended. Temperature surveys made with thermocouples peened into the specimen at various depths and taped to the surface of the reduced section showed less than 1 F. variation at the end of the 10-min. period. The same differential could be found at the end of the 1-hr. heating period, so the shorter time was used. Check tests made using both 10-min. and 1-hr. heating periods gave identical results on a number of materials. A pyrometer controlled the temperature in the windings of a resistance-wound steel-tube furnace. The temperature of the specimen was measured by means of two iron-constantan thermocouples taped to the reduced section of the specimen and protected from the ambient atmosphere by the asbestos tape. The temperature variation and accuracy of measuring equipment were well within the specified A.S.T.M. limits.

The rate of straining was controlled by the cross-head speed. The rate of cross-head travel was 0.05 in. per minute to the yield strength, and 0.25 in. per minute from this point to rupture. The specimen was held momentarily at a constant load after reaching the yield strength while the extensometer was disconnected. Strain was recorded autographically by an O. S. Peters Model No. PSH-8 high-temperature extensometer. The accuracy of the extensometer is satisfactory for yield strength determinations.

The equipment for creep testing is shown in Figs. 1 and 2. Methods of test conform to those given in the Standard Recommended Practice for Conducting Long-Time High-Temperature Tension Tests of Metallic Materials (E 22 – 41).[6] Two methods of strain measurement were used, depending on whether the test was continued to rupture, or not. For creep-to-rupture at room temperature, the strain was measured on the specimen with a dial gage accurate to 0.001 in. At high temperature, the movement of the bottom shackle was measured with the same gage. The value obtained from the shackle movement was converted to strain by dividing by 2⅜. The parallel section between the fillets of the test bar is 2¼ in. This method is reliable in giving the general shape of the creep curve, but the absolute values obtained are an approximation. The elongation in 2 in. is obtained from the specimen after rupture.

The loading mechanism for all the creep tests was identical. The frames used for creep-to-rupture are bolted securely to large blocks of concrete to minimize the reaction when a specimen breaks. For creep tests where precise measurement of strain is required, the frames are mounted on rubber in compression to control vibration. A slight amount of vibration is still noted in making strain measurements. Most of this is in the movement of the parts of the extensometer system and is not accompanied by a similar variation in stress. This was verified by the use of SR-4 electric strain gages on the bar with no change in strain being read on a cathode-ray oscilloscope.

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[4] 1944 Book of A.S.T.M. Standards, Part I, p. 994.

[5] *Ibid.*, p. 962.

[6] 1944 Book of A.S.T.M. Standards, Part I, p. 1001.

Loading is by a lever system calibrated by beam and poise up to 600 lb. and with an Amsler calibration box at 5000 and 10,000 lb. All calibrations showed the system accurate within 1 per cent. Universal joints and knife edges are used to help secure axial alignment.

Heating is by electrical resistance-wound steel-tube furnaces. These are suspended from the frames and are closed on top to prevent chimney action. Temperatures are controlled by means of a pyrometer with the hot junction of the thermocouple located in the windings of the furnace. Thermal variation during the control cycle is thus held to a minimum by controlling at the source of heat.

Since the temperature of the specimen is less than the pyrometer settings, the pyrometer must be adjusted to give the desired specimen temperature. This temperature is measured by means of a semi-precision potentiometer and an iron-constantan thermocouple taped to the specimen. The thermocouple wire is calibrated by the manufacturer. The reference temperatures are read to 0.2 C. in an insulated box containing the connector block. This temperature-measuring system was checked against a second system consisting of a calibrated thermocouple, ice-bath reference junction, and an L and N type K potentiometer. Results by both methods checked within 0.3 F. Maximum temperature variation due to the control cycle is 0.3 F. The temperature variation is less than 3 F. from the nominal test temperature over the gage length.

The room temperature has been maintained at 95 F., except for several short periods of time.

One extensometer system is shown in Fig. 2. A telescope and scale are used to measure the rotation of two mirrors fastened to rollers between extensions to collars on the reduced section of the specimen. The accuracy of the system was checked at room temperature by calibration against two Tuckerman optical strain gages reading to 0.000004 in. which had been calibrated with an accuracy of 0.2 per cent. They were also checked against two SR-4 strain gages. Modulus variations by all methods did not exceed 100,000 psi. in 6,500,000 psi. The average variation in strain obtained by any system was less than 1 per cent. The sensitivity of the optical system is excellent. By using two gages, the effects of eccentric loading are minimized.

A second extensometer, not shown, makes use of a double mirror system. The rotation of a lozenge is measured by a Tuckerman autocollimator. The accuracy of this method is better than the telescope and scale arrangement and the sensitivity is about the same.

The creep-to-rupture tests were continued to failure with a limit of about 1000-hr. duration. A semilog plot of stress *versus* time-to-rupture (log) was used to determine the load for rupture at a given time. Other values obtained are the elongation at rupture and the time-to and elongation at the beginning of the third-stage creep. The latter values were obtained at two stress levels for a given material and temperature since only selected curves were plotted.

On creep tests made with precision extensometers, where the creep is something less than failure, the load is applied in increments with concurrent measurement of strain. The stress-strain curve is plotted and the strain is entered as a separate item called the initial extension. The extension-time curve is plotted on rectangular coordinate paper. From this curve, creep extensions and creep rates are determined. A log-log plot of stress *versus* creep extension is prepared. The load necessary for any given extension is obtained from this graph. Creep rates are given between 750 and 1000 hr. if the second stage obtains. Otherwise, a note is made if the test was in the first stage, and the time to the third stage is given if the third stage was reached before the completion of the test.

Extensions obtained at the end of 100 hr. are included to compare with values at 1000 hr.

TABLE II.—SHORT-TIME TENSILE PROPERTIES.

Material	Temperature, deg. Fahr.	Tensile Strength, psi.	Yield Strength, psi.	Elongation in 2 in., per cent
SAND CASTINGS				
C-S	95	24 000	18 900	0.3
	200	24 100	16 400	0.9
	300	24 100	13 500	3.6
	450	15 500	9 600	31.9
	600	9 100	5 100	60.7
C-HTS	95	40 700	22 000	3.2
	200	39 300	18 700	7.7
	250	35 300	17 600	35.0
	300	27 500	15 700	26.0
	450	15 100	9 900	41.4
	600	8 600	5 400	76.2
C-HTA	95	43 400	26 000	2.0
	200	41 300	22 000	25.0
	250	35 600	20 000	29.7
	300	28 000	18 000	35.0
	400	16 900	10 900	36.0
	500	11 300	7 400	33.0
	600	7 800	5 100	49.0
H-S	95	28 300	17 700	3.7
	200	29 600	15 100	7.5
	250	29 400	14 400	9.2
	300	26 100	12 700	32.7
	450	14 100	7 800	42.9
	600	7 800	4 400	65.2
H-HTS	95	38 700	17 800	7.6
	200	38 000	16 500	15.2
	250	33 400	15 700	19.5
	300	27 000	14 200	40.3
	450	14 900	8 600	33.2
	600	7 300	3 700	77.2
H-HTA	95	38 700	17 700	5.5
	200	36 000	17 300	11.0
	250	32 400	16 500	11.0
	300	24 500	15 000	15.0
	400	17 500	12 000	17.0
	500	12 000	8 800	15.0
	600	8 200	5 600	20.0
E10-HTA	95	19 100	....	0.5
	300	20 100	18 200	1.0
	400	21 300	18 400	....
	500	16 700	10 200	2.0
	600	15 500	7 400	10.0
DIE CASTING				
R	95	34 200	21 900	4.0
	200	32 700	21 100	5.0
	300	27 600	16 200	14.7
	450	14 600	9 800	17.0
	600	7 000	3 600	20.7
FORGINGS				
M	95	33 600	18 800	11.6
	200	23 100	13 800	25.7
	250	22 400	16 000	25.5
	300	19 900	12 000	30.7
	400	17 200	9 100	34.2
	500	10 100	5 900	87.5
	600	6 000	3 800	140.0
O-1 HTA	95	52 900	39 500	4.5
	300	31 000	20 700	30.3
EM22	95	36 400	26 000	11.5
	300	25 400	20 600	19.0
EXTRUSIONS				
M	95	39 600	30 900	7.6
	200	27 000	21 300	15.0
	250	24 400	18 800	20.2
	300	21 300	15 700	18.7
	400	18 800	11 700	25.0
	500	13 000	7 500	60.0
	600	9 000	5 400	93.0
FS-1[a]	95	39 900	28 600	16.6
	200	34 800	22 000	20.2
	250	30 600	18 300	29.0
	300	24 700	14 500	38.2
J-1	95	45 800	33 100	14.3
	200	42 300	26 400	21.0
	250	39 000	25 800	30.7
	300	30 400	20 200	40.5
	400	21 400	14 700	42.0
	500	12 900	8 200	64.0
	600	7 900	5 100	70.0
O-1 HTA	95	56 500	40 500	4.5
	200	48 500	32 100	20.0
	250	42 000	26 600	33.0
	300	33 600	21 400	41.2
	400	21 600	14 700	49.0
	500	13 600	7 800	83.0
	600	8 700	4 700	123.0
SHEET				
Ma	95	33 000	20 200	17.7
	200	24 200	16 200	31.0
	250	21 600	14 700	40.5
	300	19 300	12 700	44.0
Mh	95	36 000	27 000	10.0
	200	29 700	26 700	7.7
	250	27 300	24 500	11.7
	300	25 000	21 200	16.0

[a] 0.5-in. diameter extrusion.

If the loading rate is abnormally slow, some creep may occur. Care was taken to insure as uniform and rapid a load application as was consistent with obtaining accurate strain readings. At room temperature, and at higher temperatures with some materials, the initial extension is the critical factor, since little creep was encountered at the yield strength of the material.

The stresses for 0.1, 0.5, and 1 per cent extension in 1000 hr. are given. For periods of longer than 1000 hr. an extrapolation may be made, but the attendant uncertainties should be considered.

The specimens tested were selected from commercial material with no special processing other than normal procedure. The cast bars were cast to size in separate molds. The extrusions were mostly $\frac{3}{4}$-in. diameter solid rounds unless otherwise noted. The sheet specimens were $\frac{1}{8}$ in. thick for the limited creep, and $\frac{1}{4}$ in. thick when creep-to-rupture was measured. Forged specimens were machined from a simple forging.

Selected specimens were examined metallographically before and after failure. No change in structure other than an elongation of the grains was observed.

RESULTS

Table II is a summary of short-time tensile properties determined at room and elevated temperatures on magnesium-base alloys. In many cases creep tests were made on material from the same batch. The temperatures of testing are from 95 to a maximum of 600 F. The effect of increasing the temperature is to lower the tensile and yield strengths and increase the elongation values. The cast cerium-containing alloy has the highest properties above 400 F. The values presented are for a small number of test bars from material with reasonably typical room-temperature properties.

TABLE III.—CREEP-TO-RUPTURE.

Material	Temperature, deg. Fahr.	Stress, psi.	Hours to Third Stage	Extension Third Stage, per cent	Hours to Rupture	Extension at Rupture, per cent
SAND CASTINGS						
C-S	95	25 000	No rupture in 1000 hr.			
		26 000	Broke in loading			
	300	11 000	300	2.3	890	20.1
		12 000	168	2.3	550	20.5
C-HTS	95	37 000	720	9.6	1176	21.0
		40 000	140	7.2	265	15.6
	300	9 000	430	3.5	910	29.6
		10 000	145	2.0	354	33.7
C-HTA	95	34 000	480	7.4	1156	28.8
		40 000	115	9.0	233	26.8
	250	12 000	495	2.7	1080	30.5
		14 000	120	5.7	290	30.9
	300	8 000	405	2.5	1181	32.9
		10 000	84	2.1	288	35.7
H-S	95	27 000	No rupture in 1000 hr.			
		28 000	Broke in loading			
	300	10 000	285	3.6	660	30.2
		12 000	78	4.3	183	29.6
H-HTS	95	39 000	730	7.6	840	14.7
	300	9 000	540	4.5	915	34.0
		10 000	180	5.5	450	26.7
H-HTA	300	9 000	330	2.4	728	10.8
		10 000	90	1.4	310	11.0
FORGINGS						
O-1 HTA	300	10 000	310	3.5	794	30.5
		12 000	160	3.5	356	26.7
EM22	300	23 000	420	0.55	1258	19.3
		24 000	230	1.4	578	17.3
EXTRUSIONS						
M	95	24 000	1300	16.6	1420	21.1
		25 000	340	13.5	477	22.1
	300	12 000	680	14.7	1115	38.5
		14 000	...	....	232	41.8
FS-1	95	31 000	570	15.1	866	30.4
		32 000	330	15.9	418	28.9
	300	7 000	390	10.5	900	55.0
		8 000	180	9.5	410	54.0
J-1	95	42 000	660	8.4	1158	24.7
		44 000	210	9.1	299	21.9
	300	6 000	360	4.2	1174	71.0
		10 000	114	3.2	312	44.4
O-1 HTA	95	40 000	610	8.4	1228	24.8
		42 000	116	12.0	284	23.2
	250	12 000	180	3.2	780	50.4
		14 000	90	4.4	370	41.5
	300	8 000	300	3.5	1148	43.9
SHEET						
Ma	95	22 000	265	9.3	630	39.8
	300	9 000	625	2.9	996	64.0
		10 000	285	2.1	531	73.0
Mh	95	25 500	1190	1.6	1645	10.6
		26 000	130	0.9	290	14.5
	300	12 000	190	5.1	800	47.7
		14 000	90	3.5	176	35.0

The time and stress for rupture from the creep tests are given in Figs. 3 to 8.

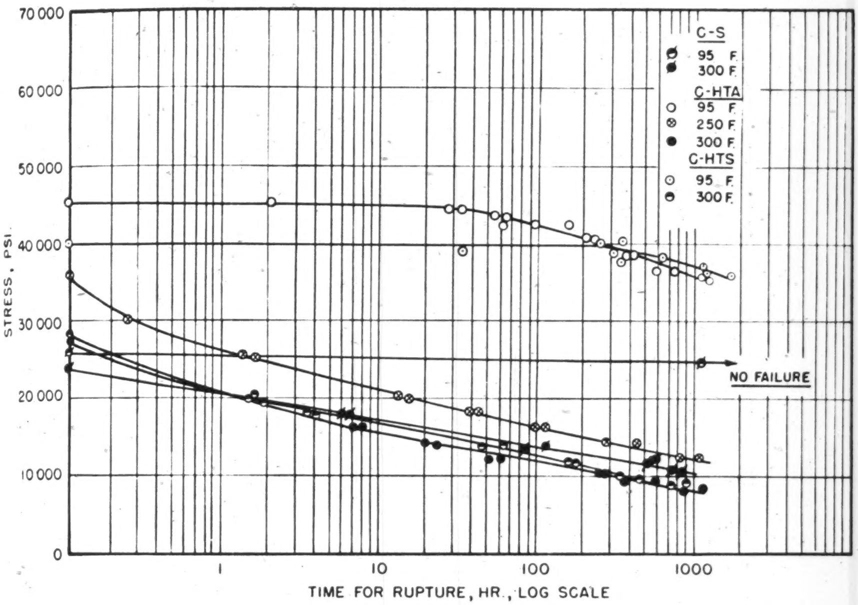

FIG. 3.—Rupture Time *versus* Stress of Alloy C-S (Sand Cast and Stabilized), C-HTS (Sand Cast, Heat-Treated, and Stabilized), and C-HTA (Sand Cast, Heat-Treated, and Aged).

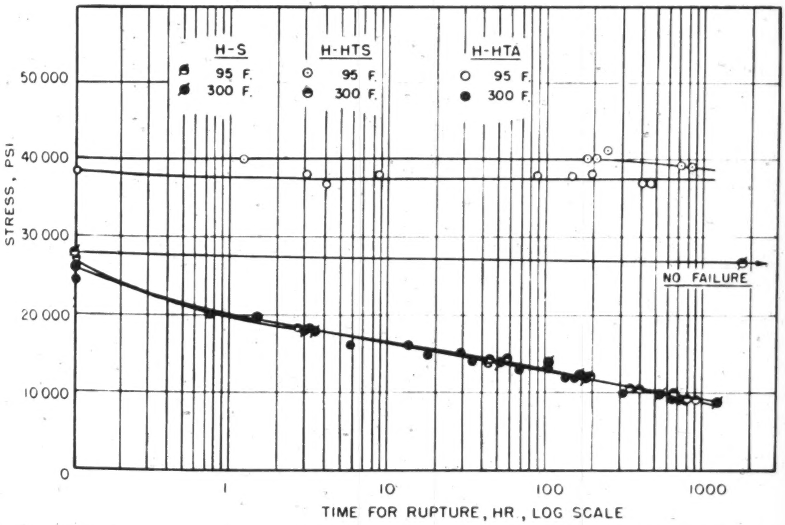

FIG. 4.—Rupture Time *versus* Stress of Alloy H-S (Sand Cast and Stabilized), H-HTS (Sand Cast, Heat-Treated, and Stabilized), and H-HTA (Sand Cast, Heat-Treated, and Aged).

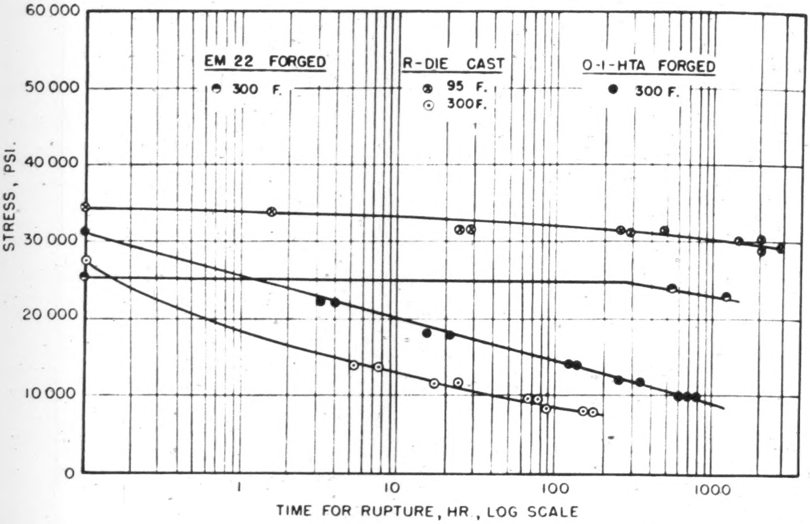

FIG. 5.—Rupture Time *versus* Stress of Alloy R (Die Cast), Alloy O-1 HTA (Forged, Heat-Treated and Aged), and Alloy EM22 (Forged).

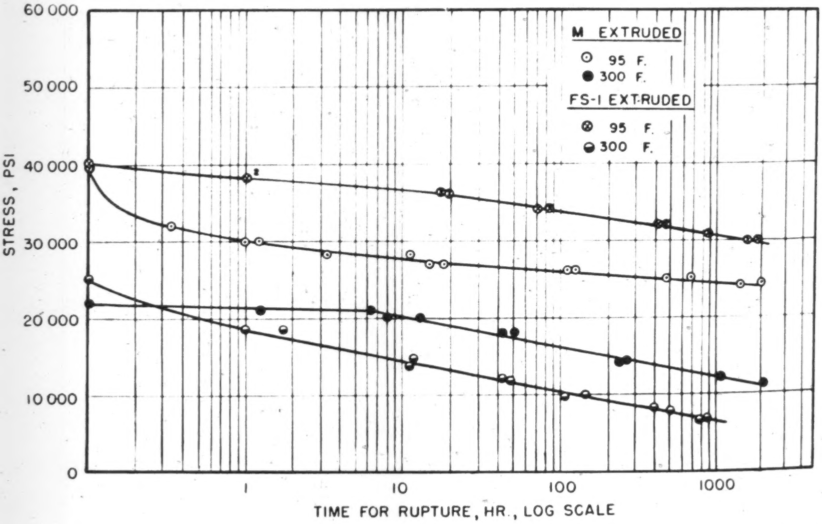

FIG. 6.—Rupture Time *versus* Stress of Alloy M (Extruded) and Alloy FS-1 (Extruded).

Duplicate specimens were run at each stress level. At 95 F. all alloys could sustain a load for 1000 hr. which was a high percentage of the short-time tensile strength. At 300 F. the stress for rupture in 1000 hr. is considerably less than the short-time tensile yield strength of the same alloys, with the exception of the cerium-containing alloys.

Table III gives data on the creep-to-rupture tests of selected test bars. Data given in addition to that on the graphs are the elongation after rupture, the time at which the test entered third-stage creep, and the strain at that point. These data were not available on all materials. On some materials, particularly the commercial cast alloys at 95 F., the ranges of the short-time strength and that at 1000 hr. actually overlapped so that one bar would break at a given stress while the next would not break in 1000 hr. The average showed that the stress for rupture in 1000 hr. was only 1000 psi. less than the short-time tensile strength.

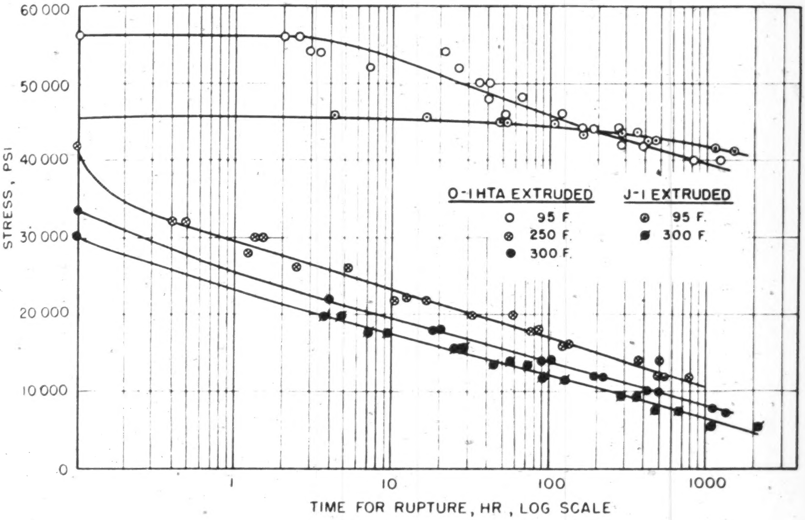

FIG. 7.—Rupture Time *versus* Stress of Alloy J-1 (Extruded) and O-1 HTA (Extruded, Heat-Treated, and Aged).

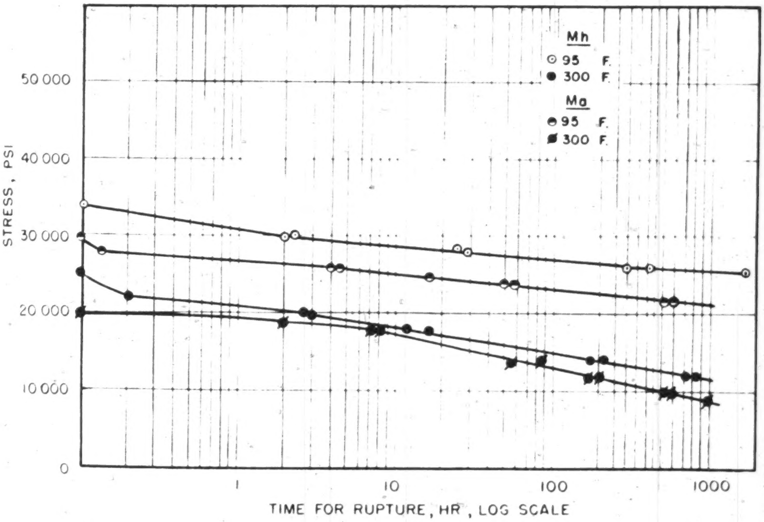

FIG. 8.—Rupture Time *versus* Stress of Alloy Ma (Annealed Sheet) and Mh (Hard-Rolled Sheet).

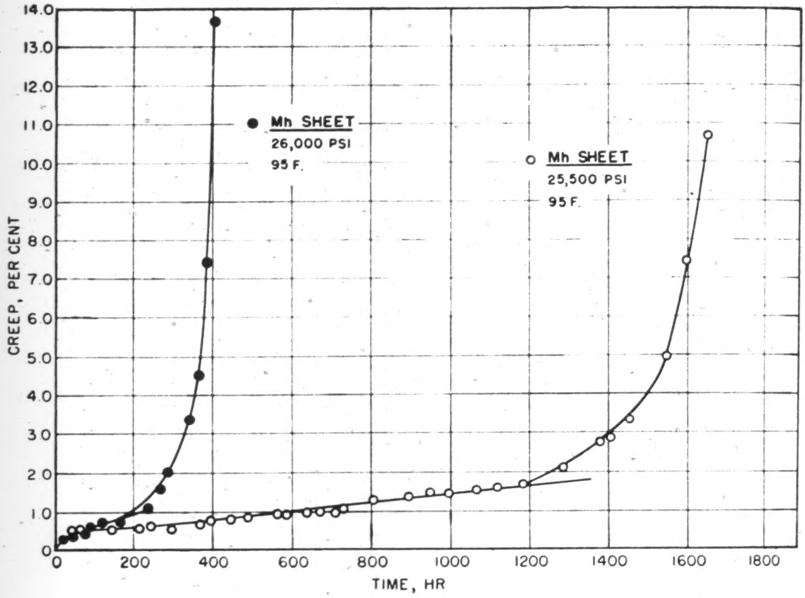

FIG. 9.—Creep-to-Rupture at 95 F. of Alloy Mh (Hard-Rolled Sheet).

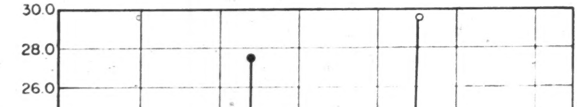

FIG. 10.—Creep-to-Rupture at 300 F. of Alloy C-HTS (Sand Cast, Heat-Treated, and Stabilized).

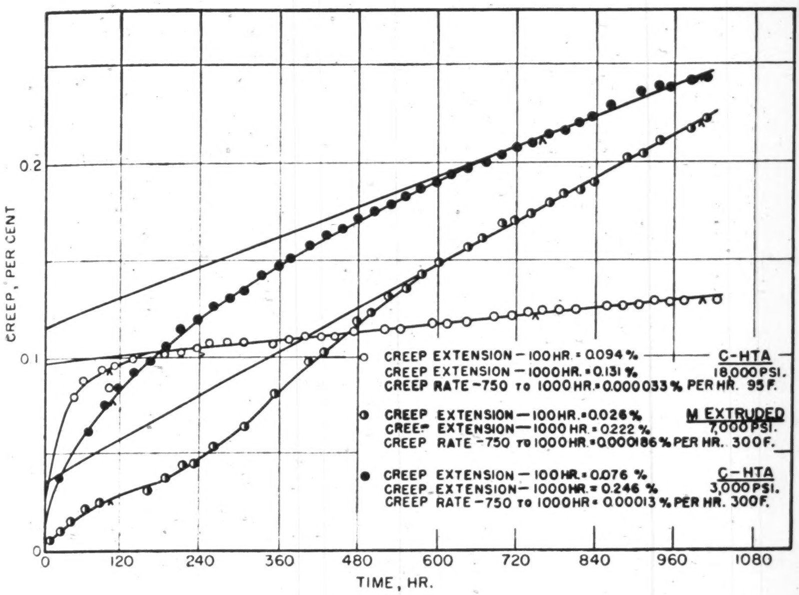

Fig. 11.—Creep Curves at 95 F., 18,000 psi., and at 300 F., 3000 psi., of Alloy C-HTA (Sand Cast, Heat-Treated, and Aged), and of Alloy M (Extruded) at 300 F. and 7000 psi.

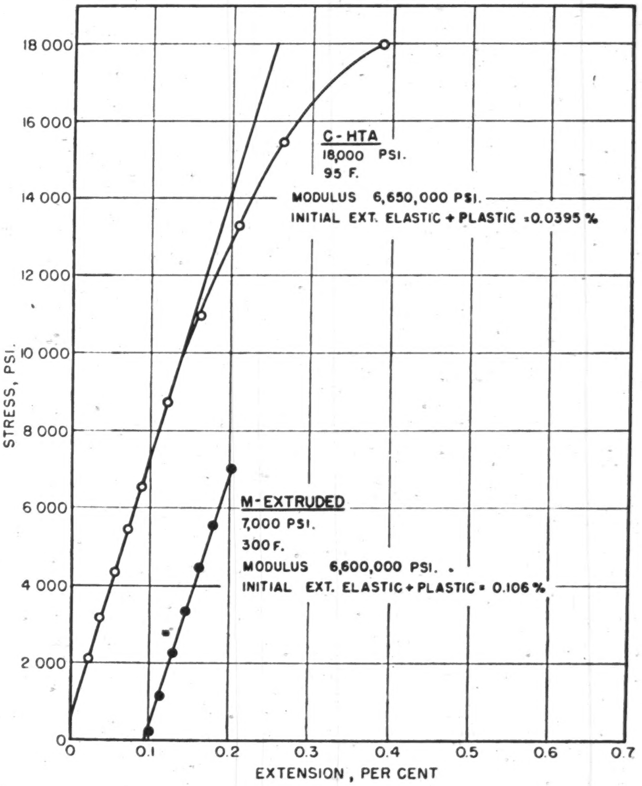

Fig. 12.—Stress-Strain Curves Obtained on Loading Alloy C-HTA (Sand Cast, Heat-Treated, and Aged) to 18,000 psi. at 95 F. and Alloy M (Extruded) to 7000 psi. at 300 F.

TABLE IV.—CREEP DATA ON INDIVIDUAL TESTS.

Material	Temperature, deg. Fahr.	Load, psi.	Initial Extension Elastic Plus Plastic, per cent	Extension by Creep, per cent		Creep Rate, 750 to 1000 hr., per cent × 10⁴
				100 hr.	1000 hr.	
SAND CASTINGS						
C-HTS	95	15 000	0.315	0.050	0.088	0.18
		17 000	0.366	0.073	0.116	0.32
		18 000	0.393	0.075	0.130	0.35
		19 000	0.428	0.084	0.152	0.40
	200	7 000	0.108	0.024	0.086	0.34
		8 000	0.130	0.072	0.148	0.64
		9 000	0.150	0.087	0.167	0.69
		11 000	0.192	0.145	0.326	1.51
	250	4 000	0.065	0.052	0.104	0.53
		5 000	0.078	0.066	0.184	0.98
		7 000	0.125	0.104	0.320	1.99
	300	2 000	0.040	0.043	0.157	1.12
		3 000	0.055	0.095	0.360	2.38
		4 000	0.070	0.133	0.550	3.88
		5 000	0.080	0.112	0.482	3.32
C-HTA	95	17 000	0.427	0.043	0.062	0.08
		18 000	0.395	0.094	0.131	0.33
		19 000	0.386	0.072	0.146	0.64
	200	5 000	0.075	0.022	0.039	0.14
		7 000	0.116	0.065	0.122	0.31
		8 000	0.130	0.079	0.175	0.80
		10 000		0.152	0.316	1.15
	300	2 000	0.035	0.056	0.139	0.36
		3 000	0.060	0.076	0.246	1.30
		4 000	0.066	0.148	0.458	2.25
		5 000	0.090	0.290	1.140	8.0
H-S	95	16 000	0.443	0.075	0.129	0.41
	200	6 000	0.095	0.048	0.082	0.32
		7 000	0.117	0.058	0.104	0.30
		9 000	0.180	0.089	0.160	0.41
	250	4 000	0.070	0.022	0.033	0.12
		5 000	0.081	0.048	0.116	0.62
		6 000	0.100	0.078	0.208	1.07
	300	2 500	0.041	0.044	0.146	0.94
		3 000	0.043	0.042	0.138	0.54
		3 000	0.050	0.132	0.273	1.09
		4 000	0.057	0.092	0.270	1.44
		5 000	0.080	0.160	0.448	1.60
H-HTS	95	16 000	0.390	0.048	0.075	0.11
		17 650	0.505	0.054	0.092	0.21
		18 000	0.379	0.074	0.123	0.25
	200	9 000	0.170	0.77	0.148	0.70
		11 000	0.217	0.093	0.194	0.69
		14 000	0.302	0.152	0.290	1.50
		15 500	0.374	0.265	0.700	3.30
	250	5 000	0.077	0.038	0.121	0.89
		5 000	0.079	0.048	0.144	0.96
		6 000	0.105	0.062	0.153	0.92
		7 000	0.117	0.073	0.164	0.80
		9 000	0.160	0.110	0.250	1.00
		10 000	0.200	0.180	0.470	2.80
		12 500	0.283	0.340	1.410	3rd stage at 770 hr
	300	2 000	0.033	0.036	0.132	0.50
		3 000	0.055	0.072	0.278	1.60
		4 000	0.072	0.098	0.400	2.64
		6 000	0.120	0.210	0.960	6.45
		7 000	0.112	0.175	1.250	11.90
H-HTA	95	16 000	0.372	0.043	0.063	0.18
		17 000	0.415	0.044	0.071	0.22
		18 000	0.483	0.056	0.096	0.34
	200	8 000	0.125	0.040	0.085	0.26
		10 000	0.178	0.068	0.117	0.34
		11 000	0.185	0.115	0.220	0.68
	300	2 000	0.032	0.047	0.184	1.04
		3 000	0.045	0.076	0.270	1.52
		4 000	0.077	0.130	0.448	2.60
		5 000	0.092	0.167	0.682	4.60
E10-HTA	300	20 000	0.555	0.078	0.174	1.07
	400	8 000	0.130	0.029	0.130	0.88
		10 000	0.170	0.036	0.254	3rd stage at 670 hr
	500	3 500	0.049	0.035	0.102	0.55
		4 500	0.090	0.084	0.224	1.28
	600	1 500	0.020	0.055	0.340	3.35
		2 500	0.047	0.230	Over 3.0% at 550 hr.	3rd stage at 100 hr.

TABLE IV—*Continued.*

Material	Temperature, deg. Fahr.	Load, psi.	Initial Extension Elastic Plus Plastic, per cent	Extension by Creep, per cent		Creep Rate, 750 to 1000 hr. per cent × 10^4
				100 hr.	1000 hr.	
			FORGINGS			
O-1 HTA	250	4 000	0.065	0.072	0.252	1.64
		6 000	0.102	0.088	0.302	2.00
	300	2 080	0.026	0.132	0.421	1.60
		2 500	0.039	0.119	0.404	2.10
		3 000	0.060	0.155	0.466	2.30
		4 000	0.073	0.173	0.653	3.70
		5 000	0.070	0.185	0.923	6.58
	350	4 000	0.061	0.527	2.675	3rd stage at 600 hr.
		5 000	0.063	0.570	11.700	3rd stage at 140 hr.
EM22	300	18 000	0.307	0.042	0.114	3rd stage at 560 hr.
		20 000	0.344	0.044	0.270	3rd stage at 240 hr.
			EXTRUSIONS			
M	95	14 000	0.220	0.016	0.079	0.66
		15 000	0.242	0.026	0.164	0.45
		16 000	0.262	0.149	0.337	0.27
	200	12 000	0.199	0.023	0.033	0.01
		14 000	0.215	0.124	0.127	0.03
		15 000	0.237	0.210	0.216	0.01
	250	10 000	0.150	0.011	0.027	0.40
		11 000	0.171	0.014	0.191	3rd stage at 720 hr.
		13 000	0.200	0.080	2.580	3rd stage at 360 hr.
		14 000	0.240	0.250	5.580	3rd stage at 240 hr.
	300	5 000	0.090	0.016	0.080	0.69
		6 000	0.094	0.020	0.133	1.03
		6 000	0.097	0.017	0.076	0.56
		7 000	0.106	0.026	0.222	1.86
		8 000	0.122	0.040	0.833	6.96
J1	95	20 000		0.053	0.064	0.34
		20 000	0.317	0.034	0.074	0.15
		20 000	0.295	0.047	0.085	0.29
		22 000	0.340	0.048	0.086	0.22
		23 000	0.360	0.048	0.080	0.32
O-1 HTA	95	18 000	0.273	0.056	0.112	0.44
		20 000	0.307	0.066	0.151	0.83
		20 000	0.310	0.060	0.152	0.60
			SHEET			
Ma	95	13 000	0.210	0.010	0.020	0.09
		13 200	0.215	0.019	0.044	0.24
		14 000	0.255	0.123	0.630	2.75
	200	11 000	0.190	0.010	0.030	0.21
	250	7 000	0.111	0.011	0.079	0.69
		8 000	0.134	0.015	0.117	1.20
	300	4 000	0.062	0.041	0.274	2.60
		5 000	0.070	0.057	0.580	7.30

Typical creep-to-rupture graphs are shown in Figs. 9 and 10. They are presented to show the type of curve obtained at both 95 and 300 F. The tests which ran only a few hundred hours showed little second-stage creep. Creep-to-rupture is frequently referred to as stress rupture.

The creep-test data where rupture did not occur are listed in Table IV. Duplicate tests were seldom run at each stress level. Stress-strain curves were obtained from the loading operation and the extension entered in the fourth column. The creep extension is given as a separate item. Typical creep curves are shown in Fig. 11, and their accompanying stress-strain curves are given in Fig. 12. The loading curve for alloy C-HTA at 3000 psi. is not shown because the stress was too low to obtain enough points to give a good graph.

It will be noted in Fig. 11 that M alloy at 300 F. shows a somewhat different type of creep curve in that the first portion has an inverse bend that is completed in about 500 hr. After 500 hr. the curve is normal.

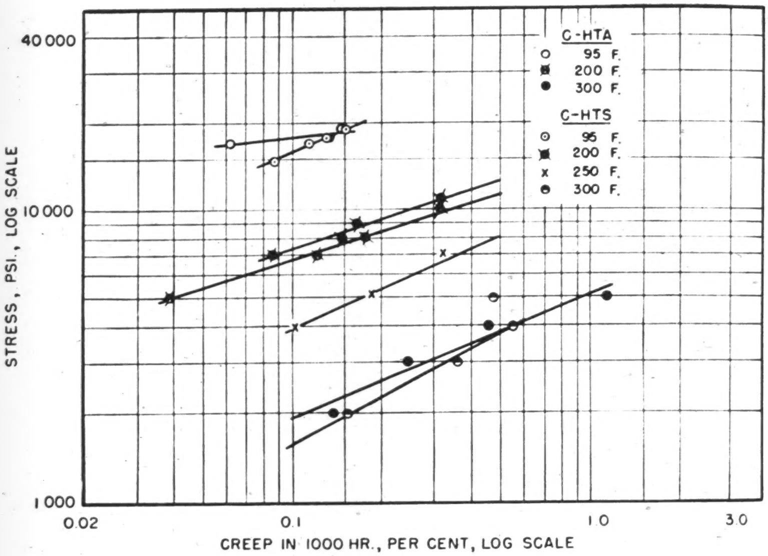

FIG. 13.—Creep Extensions at 1000 hr. *versus* Stress of Alloy C-HTS (Sand Cast, Heat-Treated and Stabilized) and C-HTA (Sand Cast, Heat-Treated, and Aged).

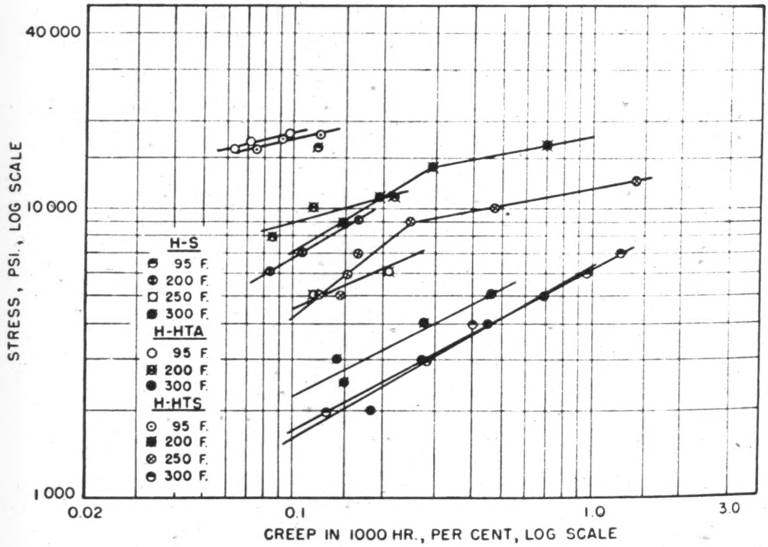

FIG. 14.—Creep Extensions at 1000 hr. *versus* Stress of Alloy H-S (Sand Cast and Stabilized), H-HTS (Sand Cast, Heat-Treated, and Stabilized), and H-HTA (Sand Cast, Heat-Treated, and Aged).

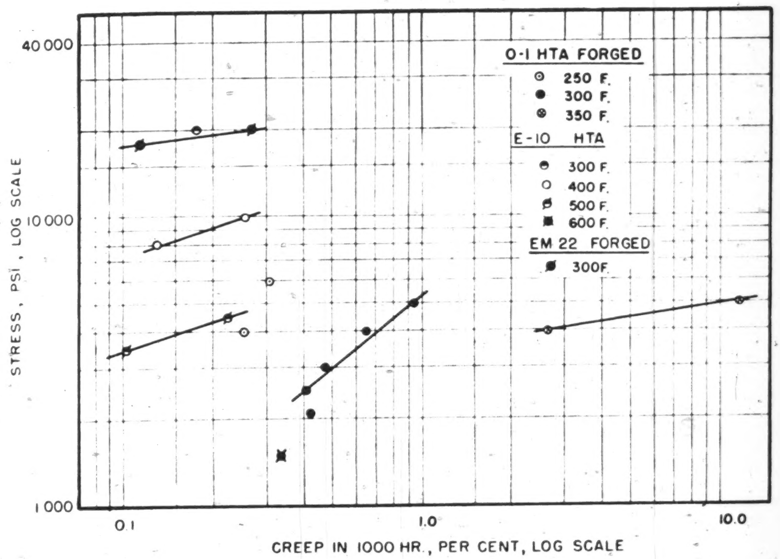

FIG. 15.—Creep Extensions at 1000 hr. *versus* Stress of Alloy EM22 (Forged), Alloy E10-HTA (Sand Cast, Heat-Treated, and Aged), and Alloy O-1 HTA (Forged, Heat-Treated, and Aged).

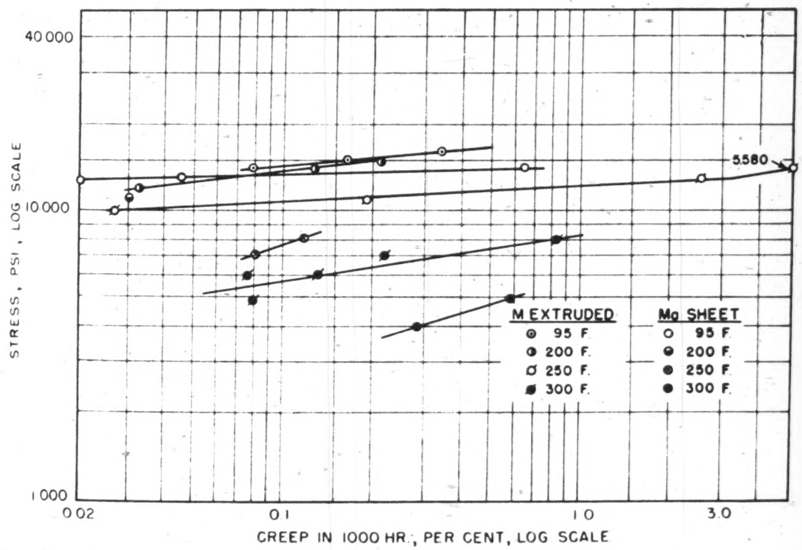

FIG. 16.—Creep Extensions at 1000 hr. *versus* Stress of Alloy M (Extruded) and Ma (Annealed Sheet).

The creep extensions at 1000 hr. are plotted against stress on a log-log scale in Figs. 13 through 16. From these are obtained the stresses for 0.1, 0.5, and 1 per cent creep in 1000 hr. given in Table V. Two values for 5 per cent extension are given. The data on some materials are meager. The values given in Table V may be changed slightly as more tests are run.

TABLE V.—SUMMARY OF CREEP RESULTS WITHOUT RUPTURE.

Material	Temperature, deg. Fahr.	Stress for 0.1 per cent Extension, psi.		Stress for 0.5 per cent Extension in 1000 hr., psi.	Stress for 1 per cent Extension in 1000 hr., psi
		100 hr.	1000 hr.		
		SAND CASTINGS			
C-HTS	95	21 800	16 000	....	....
	200	9 400	7 400	12 800	....
	250	6 900	3 900	8 100	....
	300	3 400	1 500	3 800	....
C-HTA	95	19 000	17 900	....	....
	200	8 600	6 800	11 200	....
	300	3 100	1 900	3 800	....
H-S	95	....	....	....	....
	200	9 600	6 800	....	....
	250	6 500	....	....	....
	300	4 200	2 200	5 300	....
H-HTS	95	20 100	17 400	....	....
	200	11 000	7 100	15 400	17 500
	250	8 600	4 200	10 200	11 700
	300	4 100	1 700	4 200	6 200
H-HTA	95	22 000	18 200	....	....
	200	11 000	8 900	....	....
	300	3 500	1 600	4 200	6 300
E10-HTA	400	....	7 300	....	....
	500	4 800	3 400	....	....
	600	1 800	....	....	....
		FORGINGS			
O-1HTA	300	....	....	3 000	5 300
	350	....	....	....	4 400[a]
EM22	300	....	17 800	....	....
		EXTRUSION			
M	95	16 800	14 300	16 600	....
	200	13 900	13 800	....	....
	250	13 000	10 700	11 800	12 200 14 000[a]
	300	....	5 700	7 400	8 200
		SHEET			
Ma	95	13 900	13 500	14 000	....
	200	....	....	....	....
	250	....	7 600	....	....
	300	....	....	4 800	....

[a] Stress for 5 per cent extension.

Table VI is a summary of the short-time tension and creep data assembled for ready comparison.

DISCUSSION

The main purpose in publishing these data is to make available to engineers usable information for designing parts which will operate at elevated temperature. Even though the data may not be directly applicable, they can probably be used in a comparative and qualitative way. Study of these data reveals a number of interesting points.

Both the yield strength and the tensile strength can be used in design. To compare with these values we have the stress for a certain deformation, or the stress-to-rupture under creep-loading conditions. The data for all these properties appear in Table VI

At 95 F., the stress-to-rupture is above the yield strength for nearly all the alloys and is almost as high as the tensile strength for many of them. Sand castings deform but a small amount in 1000 hr. at the yield strength. Data are available for only one wrought alloy, M, which is the lowest of the commercial wrought alloys in respect to static properties. The limiting stresses for creep conditions on this material, at this temperature, appear to be lower than the yield strength.

The data at 200 F. are limited, but some conclusions may be drawn. Sand castings may deform 1 per cent in creep in 1000 hr. at the yield strength. It is probable that the rupture values for castings are above the yield strength. Although the yield strength of extruded alloy M is lower than at 95 F., the stress for 0.1 per cent creep remains the same.

At 250 and 300 F., the yield strength appears to be higher than the highest creep stress measured—the stress-to-rupture—except for the cerium-containing alloys. The yield strength is still a

TABLE VI.—SUMMARY OF SHORT-TIME TENSION AND 1000-HR. CREEP DATA.

Material	Stress for 0.1 per cent Creep, psi.	Stress for 0.5 per cent Creep, psi.	Stress for 1.0 per cent Creep, psi.	Yield Strength, psi.	Stress for Rupture, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Elongation in 2 in. for Rupture at 1000 hr., per cent
95 F.								
SAND CASTINGS								
C-S	....	....	....	18 900	25 000	24 000	0.3	....
C-HTS	16 000	....	....	22 000	37 300	40 700	3.2	20.0
C-HTA	17 900	....	....	26 000	35 500	43 400	2.0	29.7
H-S	....	....	....	17 700	27 000	28 300	3.7	....
H-HTS	17 400	....	....	17 800	38 800	38 700	7.6	15.0
H-HTA	18 200	....	....	17 700	37 400[a]	38 700	5.5	....
DIE CASTING								
R	....	....	....	21 900	31 000	34 200	4.0	6.8
EXTRUSIONS								
M	14 300	16 600	....	30 900	24 300	39 600	7.6	22.2
FS-1	....	....	....	28 600	30 700	39 900	16.6	29.8
J-1	....	....	....	33 100	42 400	45 800	14.3	23.4
O-1 HTA	....	....	....	40 500	40 000	56 500	4.5	25.2
SHEET								
M a	13 500	14 000	....	20 200	21 700	33 000	17.7	41.5
M h	....	....	....	27 000	25 700	36 000	10.0	15.0
200 F.								
SAND CASTINGS								
C-HTS	7 400	12 800	....	18 700	....	39 300	7.7	....
C-HTA	6 800	11 200	....	22 000	....	41 300	25.0	....
H-S	6 800	....	....	15 100	....	29 600	7.5	....
H-HTS	7 100	15 400	17 500	16 500	....	38 000	15.2	....
H-HTA	8 900	....	....	17 300	....	36 000	11.0	....
EXTRUSION								
M	13 800	....	....	21 300	....	27 000	15.0	....
250 F.								
SAND CASTINGS								
C-HTS	3 900	8 100	....	17 600	....	35 300	35.0	....
C-HTA	....	....	....	20 000	11 900	35 600	29.7	32.0
H-HTS	4 200	10 200	11 700	15 700	....	33 400	19.5	....
EXTRUSIONS								
M	10 700	11 800	12 200	18 800	....	24 400	20.2	....
O-1 HTA	....	....	....	26 600	11 200	42 000	33.0	45.0
SHEET								
M a	7 600	....	....	14 700	....	21 600	40.5	....
300 F.								
SAND CASTINGS								
C-S	....	....	....	13 500	10 700	24 100	3.6	25.5
C-HTS	1 500	3 800	....	15 700	8 800	27 500	26.0	30.0
C-HTA	1 900	3 800	....	18 000	8 000	28 000	35.0	37.8
H-S	2 200	5 300	....	12 700	9 300	26 100	32.7	30.3
H-HTS	1 700	4 200	6 200	14 200	8 800	27 000	40.3	34.0
H-HTA	1 600	4 200	6 300	15 000	8 300	24 500	15.0	11.1
DIE CASTING								
R	....	....	....	16 200	8 000[b]	27 600	14.7	....
FORGINGS								
O-1 HTA	....	3 000	5 300	20 700	9 300	31 000	30.3	....
EM22	17 800	....	....	20 600	23 200	25 400	19.0	19.0
EXTRUSIONS								
M	5 700	7 400	8 200	15 700	12 200	21 300	18.7	48.0
FS-1	....	....	....	14 500	6 800	24 700	38.2	52.0
J-1	....	....	....	20 200	7 000	30 400	40.5	57.2
O-1 HTA	....	....	....	21 400	8 300	33 600	41.2	42.8
SHEET								
M a	....	4 800	....	12 700	8 800	19 300	44.0	76.0
M h	....	....	....	21 200	11 500	25 000	16.0	50.2

[a] Stress for rupture in 500 hr.

[b] Stress for rupture in 200 hr.

conservative design criterion for them. Wrought M alloy has the best creep properties in the field of commercial alloys, at these temperatures.

When rupture does occur after long periods of loading, the failure is of a ductile nature. This means that complete failure will not occur without prior warning from relatively large deformations. The loss of ductility often observed in other materials under these conditions has not been observed in magnesium-base alloys.

Use has often been made of relatively short-time creep tests to measure creep properties.[7] In this laboratory the stress for 0.1 per cent creep extension in 100 hr. has been determined[3] in order more quickly to determine creep behavior on a large number of materials. Inspection of Table V shows that this stress rates the materials in much the same order as does the limit for 1000-hr. testing. No comparison of the limits was made for higher values of extension.

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[7] A. Beck, "The Technology of Magnesium and Its Alloys," F. A. Hughes, Ltd., London (1940).

DISCUSSION

MR. P. G. McVETTY.[1]—Reference to Fig. 14 of the paper shows a break in some of the log-log curves used to express the relation between stress and the resulting creep in 1000 hr. This type of curve is found frequently and it raises a question as to whether tests at lower stresses would have shown a similar break in the other curves. To those of us who have occasion to extrapolate such curves into the low-stress field to obtain design information, this means that an estimate of the stress which will produce a specified low value of allowable creep may be much too high if it is based on the straight line log-log curves in Figs. 13, 14, 15, and 16.

In many cases it has been found that a hyperbolic sine curve fits the test data better than the power function represented by a straight line log-log plot and it leads to a more conservative estimate of stress for low values of allowable creep within the expected service life. This subject was discussed in some detail in 1943[2,3] and it has an important bearing upon the interpretation of creep test data. I should like to ask the authors whether they have tried to fit hyperbolic sine curves to their data.

MESSRS. A. A. MOORE AND J. C. McDONALD (*authors' closure by letter*).—The hyperbolic-sine relation between stress and extension was applied to the points shown on Fig. 14. The points did not lie on a straight line on the log-log plot. Using the extreme points as a basis of calculation according to the method suggested by Mr. McVetty in his paper "Creep of Metals at Elevated Temperatures; the Hyperbolic-Sine Relation Between Stress and Creep Rate,"[4] a slightly curved line was obtained which does not fit the other points. This method would be slightly better than a single straight line, but the curve is not great enough to follow the abrupt change in slope.

We have no data at lower stresses than those shown on the published figures so are unable to say whether some curves might be obtained which could be well fitted by the hyperbolic-sine relation.

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[1] Mechanical Engineer, Research Labs., Westinghouse Electric Corp., East Pittsburgh, Pa.

[2] P. G. McVetty, "Creep of Metals at Elevated Temperatures—The Hyperbolic Sine Relation Between Stress and Creep Rates," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 65, pp. 761–769 (1943).

[3] A. Nadai and P. G. McVetty, "Hyperbolic Sine Chart for Estimating Working Stresses of Alloys at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 735 (1943).

[4] P. G. McVetty, "Creep of Metals at Elevated Temperatures; the Hyperbolic-Sine Relation Between Stress and Creep Rate," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 65, pp. 761–767 (1943).

CREEP TESTS ON SOME EXTRUDED LEAD AND LEAD-ALLOY SLEEVES AND TAPES*

By G. R. Gohn,[1] S. M. Arnold,[1] and G. M. Bouton[1]

Synopsis

Creep tests covering a period of approximately 8 yr. on specimens from 16 commercial lead cable sleeves of 6 different compositions and from 14 experimental lead-alloy tapes are discussed in this paper. The data show that Chemical lead sleeves were more resistant to creep than lead-tin or lead-antimony sleeves at low stresses but not at high stresses. For the tape specimens, a similar relationship was found between Chemical lead and all of the lead alloys except the high-calcium alloys which were superior to Chemical lead in creep resistance at all stresses. High-purity, fire-refined lead, which contains smaller percentages of minor constituents than Chemical lead, was inferior to Chemical lead at all stresses.

To minimize the entrance of moisture through any defects in cable sheath and to operate alarm systems to indicate the development of sheath breaks, the Bell System, in the late 1920's, adopted the practice of maintaining gas pressure in many of its lead-covered cables. With the introduction of this procedure, the creep strength of the sleeving materials became an important factor. These sleeves (Fig. 1) are extruded, cylindrical tubes used to cover the spliced ends of two or more cables and are joined to the sheaths by means of wiped solder joints. Because the cables are subjected to a maximum gas pressure of approximately 9 psi., the hoop stresses in the sleeves may be as great as 175 psi. Since the literature contained only meager data relative to the creep strength of lead and its alloys at stresses below 200 psi., this study was undertaken to determine the creep characteristics of various grades of lead and of lead alloys as an aid in the selection of a suitable sleeving material and to establish maximum allowable stresses.

Review of Previous Work

While there is a considerable amount of published data on the creep characteristics of lead alloys, most of these data cover stresses in excess of 200 psi. for relatively short periods of time. Most of these data have been obtained since 1928 when the failure of a number of pressurized, oil-filled power cables stimulated interest in the creep of lead and its alloys. The various reports in the literature can be divided into three groups: (1) those dealing with long-time tension tests on various grades and alloys of lead, (2) those dealing with bursting tests on pipe or sheath samples, and (3) those dealing either with the mechanism of creep or the effect of minor impurities. A brief summary of this work is given in the following paragraphs.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] Members of Technical Staff, Bell Telephone Laboratories, Inc., New York, N. Y.

Extensive work, undertaken by the Utilities Research Committee in co-operation with the University of Illinois Engineering Experiment Station, is reported in a series of papers by Moore and his co-workers (**1**, **2**, **3**, **4**, **5**).[2] Most of this work was done on tension specimens cut from flattened sheath. Based upon these studies, Moore and Dollins (**5**) suggested in their latest report that the tensile stress required to produce failure in lead alloys at any given time can be computed from the formula

$$S = S_1 (1 - c \log t)$$

where S is the rupture stress in psi. for time t in hours, while S_1 and c are constants determined experimentally. Using this formula, they have extrapolated their 10,000-hr. stress-rupture data to estimate the creep strength of some 35 different sheaths. The extrapolated values indicate that the tensile stress which will produce failure in 100,000 hr. varies from 105 psi. for common lead to 900 psi. for a lead-calcium-magnesium-tin alloy. However, since their plotted data do not fall on a straight line, extrapolated values based upon the use of this formula are questionable. These investigators also found no significant difference between the results of transverse and longitudinal creep tests on lead alloys. At the longest period of time for which these authors present data (10,000 hr.), the alloys containing calcium or 0.75 per cent antimony had the highest rupture stress. Of the unalloyed leads, Chemical lead had the highest rupture stress after 10,000 hr.

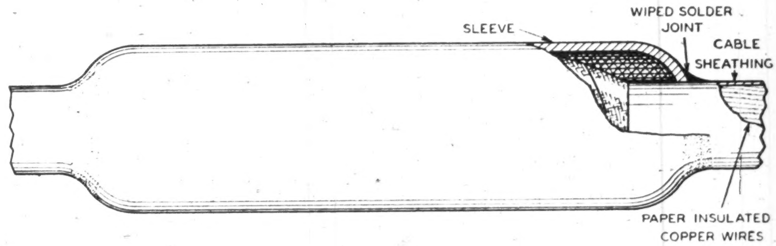

FIG. 1.—Section of Spliced Lead-Covered Cable Showing Sleeve in Place.

Most of the creep tests made at the University of Illinois employed specimens from flattened sheath. On the other hand, Phelps, Gates, and Kahn (**6**) and Bassett and Snyder (**7**) were primarily concerned with bursting tests on sheath, although the latter authors made some creep tests on tension specimens. From their tests, most of which involved intermittent loading at elevated temperatures,—conditions simulating those found in oil-filled power cables—Bassett and Snyder (**7**) concluded that at some low stress such as 400 psi., all types of lead and lead alloys, regardless of their tensile strength as normally expressed, would fail in approximately the same length of time.

Moore, Dollins, and Craig (**4**) have shown that their long-time tension tests on specimens can be correlated with the bursting tests made by Phelps, Gates, and Kahn on short lengths of the same sheaths. They therefore concluded that, for the sheaths studied, the tension-creep test is just as reliable as the more expensive bursting test in evaluating creep characteristics. The data presented by Phelps, Gates, and Kahn showed that, on the basis of bursting tests made at a temperature of 110 F., lead corresponding to Chemical lead had a higher bursting strength after 1500 hr. than did high-bismuth lead or copper-bearing lead containing 0.06 per cent bismuth.

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[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 1019.

Greenwood and his co-workers (**8, 9, 10, 11**) have shown that minor impurities materially influence the creep characteristics of lead.

Smith (**12**), studying pure lead, showed that creep, instead of being a continuing deformation of the material, may be intermittent with periods of rapid and extremely slow extension. The author reported recrystallization at high stresses but was unable to detect recrystallization at low stresses. When recrystallization was a factor, it was found that the measured rate of creep was greater than the value obtained by extrapolation.

Bibliographies covering the work of other investigators are given in the papers referred to and also in a paper on this subject by Phillips (**13**).

From the review of the literature on creep, it would appear that the greatest need is for more data at low stresses for long periods of time. The present paper is a contribution toward supplying data in this field.

MATERIALS INVESTIGATED

The materials studied in this investigation included 16 sleeves of 6 different compositions representing standard commercial alloys extruded early in 1937, and 14 experimental tapes extruded between 1933 and 1936.

The following sleeves were tested:

Sleeve	Sleeve Manufacturer	Description
Nos. 1 and 10	A	Chemical lead—0.9 per cent antimony
Nos. 3 and 9	B	Secondary lead—0.7 per cent antimony
Nos. 15[a] and 16[a]	B	Secondary lead—0.7 per cent antimony
Nos. 4 and 5	B	Secondary lead—3 per cent tin
Nos. 2 and 7	B	Secondary lead
Nos. 13 and 14	B	Detinned lead[b]
Nos. 6 and 8	A	Chemical lead
Nos. 11 and 12	B	Chemical lead

[a] Check sleeves.

[b] Recovered from lead, 3 per cent tin cable sheath returned from the field.

Included in this list are duplicate sleeves of each alloy, together with Chemical-lead sleeves from two manufacturers and two check sleeves of the 0.7 per cent antimony alloy extruded at a later date.

While conditions existing at the plant of manufacturer A during extrusion are not known, sleeves supplied by manufacturer B were extruded vertically by an inverted extrusion process. In this process the outer sizing die was held in a hollow piston and the core tube was supported at the bottom of the lead chamber. The sleeves were extruded at approximately 200 C. and allowed to

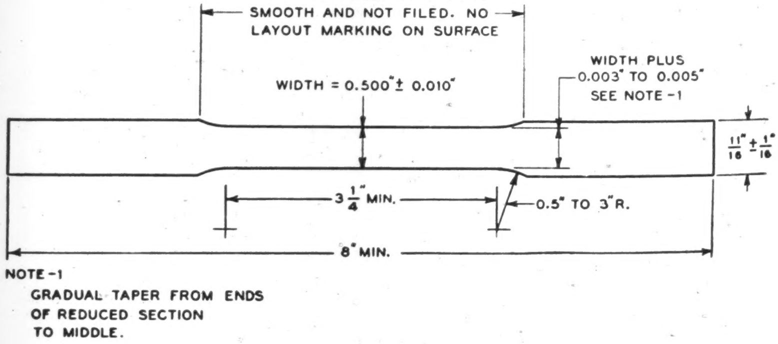
MUST BE MILLED OR GROUND SMOOTH AND NOT FILED. NO LAYOUT MARKING ON SURFACE
WIDTH = 0.500" ± 0.010"
WIDTH PLUS 0.003" TO 0.005" SEE NOTE -1
11"/16 ± 1"/16
3 1/4" MIN.
0.5" TO 3" R.
8" MIN.
NOTE -1
GRADUAL TAPER FROM ENDS OF REDUCED SECTION TO MIDDLE.

FIG. 2.—Tension and Creep Test Specimen.

cool freely in still air, reaching room temperature in about an hour.

The experimental tapes included in this study were:

Tape	Extrusion Temperature, deg. Cent.	Description
No. 68D	235	High-purity, fire-refined lead
No. 89F	279	Chemical lead
No. 84A	252	Chemical lead—0.29 per cent antimony
No. 91F	236	Chemical lead—1.0 per cent antimony
No. 29E	239	High-purity, fire-refined lead —0.02 per cent calcium
No. 63C	237	High-purity, fire-refined lead —0.027 per cent calcium
No. 76L	222	Chemical lead—0.013 per cent calcium
No. 77L	209	Chemical lead—0.024 per cent calcium
No. 74H	277	Chemical lead—0.033 per cent calcium
No. 54C	237	Chemical lead—0.036 per cent calcium
No. 55A	244	Chemical lead—0.043 per cent calcium
No. 10B	215	Chemical lead—0.05 per cent calcium
No. 60A	241	High-purity, fire-refined lead —0.06 per cent tellurium
No. 92F	229	Chemical lead—0.07 per cent tellurium

Among the tape alloys were two compositions similar to those supplied as sleeving. The range of compositions was also extended to include high-purity, fire-refined lead, and alloys containing calcium and tellurium.

The tape alloys were extruded at the Bell Telephone Laboratories in a press designed to simulate the conditions existing during the sheathing of paper-insulated telephone cables. All of the tape alloys were cooled to room temperature in about 16 hr. Although the variation in extrusion temperatures and the long cooling period may have had some effect on the creep strength, it is believed that this was secondary to the effect of the alloying constituents. The latter is of primary interest in this paper.

METHODS OF TEST AND TEST DATA

The chemical analyses of the different materials investigated are given in Table I. Conventional wet methods of analysis were used except as noted. Whereas a complete analysis was made of the sleeving alloys, the tapes were analyzed only for the major alloying constituent, since leads of established purity were used as the basis materials.

The tensile strength and the corresponding percentage elongation in 2 in. were determined on specimens cross-milled to the dimensions shown in Fig. 2. Both transverse and longitudinal specimens were taken from flattened sleeves, while longitudinal specimens alone were taken from the tapes. These specimens are similar to the standard 2-in. gage length tension test specimen shown in Fig. 2 of A.S.T.M. Standard Methods of Tension Testing of Metallic Materials (E 8–42)[3], except that the reduced section was $3\frac{1}{4}$ in. in length instead of $2\frac{1}{4}$ in. In computing tensile strengths, the cross-section of the specimens was determined from measurements made to the nearest 0.0001 in. All tension tests were made on a 600-lb. Amsler pendulum type testing machine, accurate to ± 1 per cent at any load, and operated at a head-speed of 0.250 in. per inch per minute.

To determine the effect of aging during the course of the creep tests, tension tests were made on sleeves after aging at room temperature at various periods up to 8 yr. Typical data are shown in Table II for 1 month, 6 months, and approximately 8 yr. for the sleeving alloys, and at the start of the creep tests and 8 yr. later for the tape alloys.

The creep tests were made by hanging the standard tension specimens, previously described, on specially designed racks. The racks were constructed on heavy concrete bases resting upon shock-absorbing material to dampen any extraneous vibrations. For low stresses, the loads were applied directly

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[3] 1944 Book of A.S.T.M. Standards, Part I, p. 962.

TABLE I.—CHEMICAL ANALYSIS OF LEAD AND LEAD-ALLOY SLEEVES AND TAPES.

Material	Designation	Manufacturer	Description	Type of Lead	Composition, per cent[a]										
					Antimony	Tin	Calcium	Tellurium	Silver	Copper	Arsenic	Iron	Zinc	Bismuth	Lead
Sleeve	No. 1	A	Lead-0.9% Sb	Chemical	0.91	<0.001(S)	....	....	0.0068	0.059	0.0013	0.0009	<0.003(S)	nil	Balance
	No. 10	A	Lead-0.9% Sb	Chemical	0.91	<0.001(S)	....	....	0.0072	0.059	0.0015	0.0011	<0.003(S)	0.003	Balance
	No. 3	B	Lead-0.7% Sb	Secondary[c]	0.68	0.007	....	....	0.0060	0.048	nil	0.0005	<0.003(S)	0.049	Balance
	No. 9	B	Lead-0.7% Sb	Secondary[c]	0.67	0.006	....	....	0.0061	0.048	0.0004	<0.0005	<0.003(S)	0.047	Balance
	No. 15	B	Lead-0.7% Sb	Secondary[c]	0.71	0.04(S)	....	....	0.0076	0.042	nil	0.003	<0.01	0.044	Balance
	No. 16	B	Lead-0.7% Sb	Secondary[c]	0.71	<0.001(S)	....	....	0.0054	0.042	nil	0.003	<0.01	0.063	Balance
	No. 4	B	Lead-3% Sn	Secondary[c]	0.13	2.96	....	....	<0.01(S)	0.034	0.0017	0.0006	<0.003(S)	0.057	Balance
	No. 5	B	Lead-3% Sn	Secondary[c]	0.13	3.03	....	....	<0.01(S)	0.028	0.0029	0.0011	<0.003(S)	0.037	Balance
	No. 2	B	Lead	Secondary[c]	0.17	0.018	....	....	0.0043	0.025	0.0006	0.0005	<0.003(S)	0.072	Balance
	No. 7	B	Lead	Secondary[c]	0.09	0.054	....	....	0.0049	0.027	nil	0.0011	<0.003(S)	0.086	Balance
	No. 13	B	Lead	Detinned[d]	0.03	0.050	....	....	0.0061	0.033	nil	<0.0005	<0.003(S)	0.056	Balance
	No. 14	B	Lead	Detinned[d]	0.03	0.050	....	....	0.0061	0.033	nil	<0.0005	<0.003(S)	0.058	Balance
	No. 6	A	Lead	Chemical	<0.01(S)	0.002(S)	....	....	0.0048	0.063	nil	0.0009	<0.003(S)	0.005	Balance
	No. 8	A	Lead	Chemical	<0.01(S)	0.006(S)	....	....	0.0051	0.064	nil	<0.0005	<0.003(S)	0.002	Balance
	No. 11	B	Lead	Chemical	0.01(S)	<0.001(S)	....	....	0.0054	0.064	nil	0.0011	<0.003(S)	0.003	Balance
	No. 12	B	Lead	Chemical	0.01(S)	<0.001(S)	....	....	0.0054	0.059	nil	0.0009	<0.003(S)	0.004	Balance
Pig lead	....	ASTM Requirements[a]		Chemical	....	....	....	....	0.002 to 0.020	0.040 to 0.080	....	0.002 maximum	0.001 maximum	0.005 maximum	Balance As+Sb+Sn 0.002 maximum
Tape	No. 68D	B.T.L.[b]	Lead	High-purity, fire-refined	....	....	....	....	....	....	....	....	....	....	
	No. 89F	B.T.L.	Lead	Chemical	....	....	....	....	....	....	....	....	....	....	
	No. 60A	B.T.L.	Lead-0.06% Te	High-purity, fire-refined	....	....	....	0.06	....	....	....	....	....	....	
	No. 92F	B.T.L.	Lead-0.07% Te	Chemical	....	....	....	0.07	....	....	....	....	....	....	
	No. 84A	B.T.L.	Lead-0.29% Sb	Chemical	0.29	....	....	....	....	....	....	....	....	....	
	No. 91F	B.T.L.	Lead-1.0% Sb	Chemical	1.0	....	....	....	....	....	....	....	....	....	
	No. 29E	B.T.L.	Lead-0.02% Ca	High-purity, fire-refined	....	....	0.02	....	....	....	....	....	....	....	
	No. 63C	B.T.L.	Lead-0.027% Ca	High-purity, fire-refined	....	....	0.027	....	....	....	....	....	....	....	
	No. 76L	B.T.L.	Lead-0.013% Ca	Chemical	....	....	0.013	....	....	....	....	....	....	....	
	No. 77L	B.T.L.	Lead-0.024% Ca	Chemical	....	....	0.024	....	....	....	....	....	....	....	
	No. 74H	B.T.L.	Lead-0.033% Ca	Chemical	....	....	0.033	....	....	....	....	....	....	....	
	No. 54C	B.T.L.	Lead-0.036% Ca	Chemical	....	....	0.036	....	....	....	....	....	....	....	
	No. 55A	B.T.L.	Lead-0.043% Ca	Chemical	....	....	0.043	....	....	....	....	....	....	....	
	No. 10B	B.T.L.	Lead-0.05% Ca	Chemical	....	....	0.05	....	....	....	....	....	....	....	

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[a] Standard Specifications for Pig Lead (B 29 – 43), 1944 Book of A.S.T.M. Standards, Part I, p. 855.

[b] B.T.L. = Bell Telephone Laboratories, Inc.

[c] Lead recovered from refining miscellaneous scrap.

[d] Lead recovered from lead-3 per cent tin cable sheath returned from the field.

[e] (S) = Analysis made by spectrochemical methods.

[a] Standard Specifications for Pig Lead (B 29 - 43), 1944 Book of A.S.T.M. Standards, Part I, p. 855.
[b] B.T.L. = Bell Telephone Laboratories, Inc.
[c] Lead recovered from refining miscellaneous scrap.
[d] Lead recovered from lead-3 per cent tin cable sheath returned from the field.
[e] (S) = Analysis made by spectrochemical methods.

Material	Designation	Date Extruded	Manufacturer	Description	Type of Lead	Tensile Properties of Transverse Specimens After Aging[b]								Tensile Properties of Longitudinal Specimens After Aging[b]							
						1 month		6 months		12 months		8½ yr.		1 month		6 months		12 months		8½ yr.	
						Tensile Strength, psi.	Elongation in 2 in., per cent	Tensile Strength, psi.	Elongation in 2 in., per cent	Tensile Strength, psi.	Elongation in 2 in., per cent	Tensile Strength, psi.	Elongation in 2 in., per cent	Tensile Strength, psi.	Elongation in 2 in., per cent	Tensile Strength, psi.	Elongation in 2 in., per cent	Tensile Strength, psi.	Elongation in 2 in., per cent	Tensile Strength, psi.	Elongation in 2 in., per cent
Sleeve...	No. 1	1/15/37	A	Lead-0.9% Sb	Chemical	3900	41.3	....	....	....	....	3460	38.0	3870	41.0	....	....	....	....	3680	33.0
	No. 10	1/15/37	A	Lead-0.9% Sb	Chemical	3690	40.8	....	....	....	....	3690	32.0	3630	43.6	....	....	....	....	3450	33.0
	No. 3	1/15/37	B	Lead-0.7% Sb	Secondary	3100	52.6	3120	50.4	3230	52.5	2890	42.0	3120	55.6	3110	35.0	3190	43.0	2910	46.0
	No. 9	1/15/37	B	Lead-0.7% Sb	Secondary	3120	57.5	3080	62.2	3160	50.0	3150	44.5	3120	56.9	3090	60.5	3170	56.5	3120	56.0
	No. 15	....	B	Lead-0.7% Sb	Secondary	3220	57.8	....	....	....	....	3580	53.0	3240	57.6	....	....	....	....	3630	52.0
	No. 16	....	B	Lead-0.7% Sb	Secondary	3140	56.6	....	....	....	....	3150	60.0	3170	57.4	....	....	....	....	3190	56.0
	No. 4	1/15/37	B	Lead-3% Sn	Secondary	3140	61.6	....	....	....	....	3060	56.0	3080	61.1	....	....	....	....	3000	67.0
	No. 5	1/15/37	B	Lead-3% Sn	Secondary	3320	60.8	....	....	....	....	3240	57.0	3230	53.9	....	....	....	....	3100	57.0
	No. 2	1/15/37	B	Lead	Secondary	2630	69.4	2520	70.8	2580	61.0	2450	69.0	2650	69.4	2540	65.0	2640	60.0	2530	54.0
	No. 7	1/15/37	B	Lead	Secondary	2520	77.0	2420	69.2	2490	55.0	2390	60.0	2520	73.6	2460	85.0	2540	75.0	2490	70.0
	No. 13	2/15/37	B	Lead	Detinned	2340	67.4	....	....	....	....	2200	66.0	2380	68.1	....	....	....	....	2250	69.0
	No. 14	2/15/37	B	Lead	Detinned	2370	64.9	....	....	....	....	2190	61.0	2380	67.9	....	....	....	....	2210	63.0
	No. 6	1/15/37	A	Lead	Chemical	2250	47.9	....	....	....	....	2260	48.0	2270	54.9	....	....	....	....	2260	53.0
	No. 8	1/15/37	A	Lead	Chemical	2250	53.1	....	....	....	....	2270	46.0	2260	56.1	....	....	....	....	2280	54.0
	No. 11	1/12/37	B	Lead	Chemical	2330	53.4	....	....	....	....	2300	49.0	2350	68.4	....	....	....	....	2310	60.0
	No. 12	1/12/37	B	Lead	Chemical	2290	54.3	....	....	....	....	2250	49.0	2300	61.1	....	....	....	....	2310	62.0

Material	Designation	Date Extruded	Manufacturer	Description	Type of Lead	Tensile Properties of Longitudinal Specimens at Start of Creep Tests[c]		Tensile Properties of Longitudinal Specimens after Aging 9 to 12 yr.[b]	
						Tensile Strength, psi.	Elongation in 2 in., per cent	Tensile Strength, psi.	Elongation in 2 in., per cent
Tape...	No. 68D	11/13/34	B.T.L.[a]	Lead	High-purity, fire-refined	1690	63.0	1830	79.0
	No. 89F	6/18/36	B.T.L.	Lead	Chemical	1800	66.3	1990	66.0
	No. 60A	8/21/34	B.T.L.	Lead-0.06% Te	High-purity, fire-refined	2200	55.0	2540	53.0
	No. 92F	7/ 9/36	B.T.L.	Lead-0.07% Te	Chemical	2270	50.3	2570	49.0
	No. 84A	1/15/36	B.T.L.	Lead-0.29% Sb	Chemical	2050	50.0	2180	55.0
	No. 91F	7/ 7/36	B.T.L.	Lead-1.0% Sb	Chemical	2680	39.0	3090	37.0
	No. 29E	1/15/34	B.T.L.	Lead-0.02% Ca	High-purity, fire-refined	3520	38.5	4060	36.0
	No. 63C	9/25/34	B.T.L.	Lead-0.027% Ca	High-purity, fire-refined	3390	35.0	4080	34.0
	No. 76L	2/19/35	B.T.L.	Lead-0.013% Ca	Chemical	1980	51.0	2260	52.0
	No. 77L	2/26/35	B.T.L.	Lead-0.024% Ca	Chemical	2840	42.0	3290	40.0
	No. 74H	2/ 6/35	B.T.L.	Lead-0.033% Ca	Chemical	3230	34.5	4150	32.0
	No. 54C	6/12/34	B.T.L.	Lead-0.036% Ca	Chemical	4470	32.2	5220	38.0
	No. 55A	6/14/34	B.T.L.	Lead-0.043% Ca	Chemical	4420	32.7	5170	36.0
	No. 10B	8/ 1/33	B.T.L.	Lead-0.05% Ca	Chemical	5420	29.5	6280	34.0

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[a] B.T.L. = Bell Telephone Laboratories, Inc. [b] All specimens were aged at room temperature prior to testing. [c] These tension tests were made in November, 1936.

to the specimens (Fig. 3); for high stresses a lever system was used (Fig. 4). Creep tests were made on all of the alloys at a temperature of 80 to 85 F. In addition, a few tests were made at 20 F. on three of the tape alloys to determine the effect of lower temperatures.

The creep of the specimens was determined from measurements made between bench marks scribed approximately 3 in. apart on the specimens prior to hanging. The exact distance between bench marks was determined after hanging the specimens, but prior to application of the load. For high stresses, the elongation of the specimen was measured periodically by means of dividers with a precision of ± 0.3 per cent over a 3-in. gage length; for low

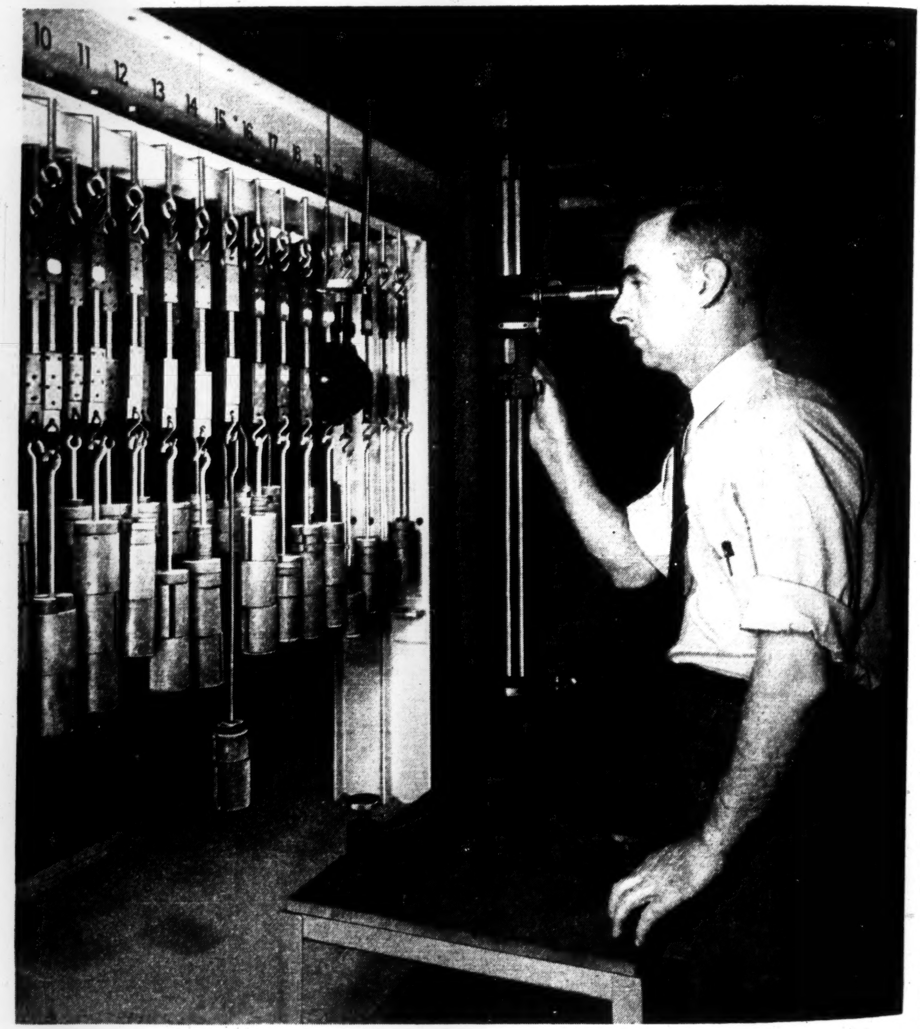

FIG. 3.—Creep Rack and Cathetometer.

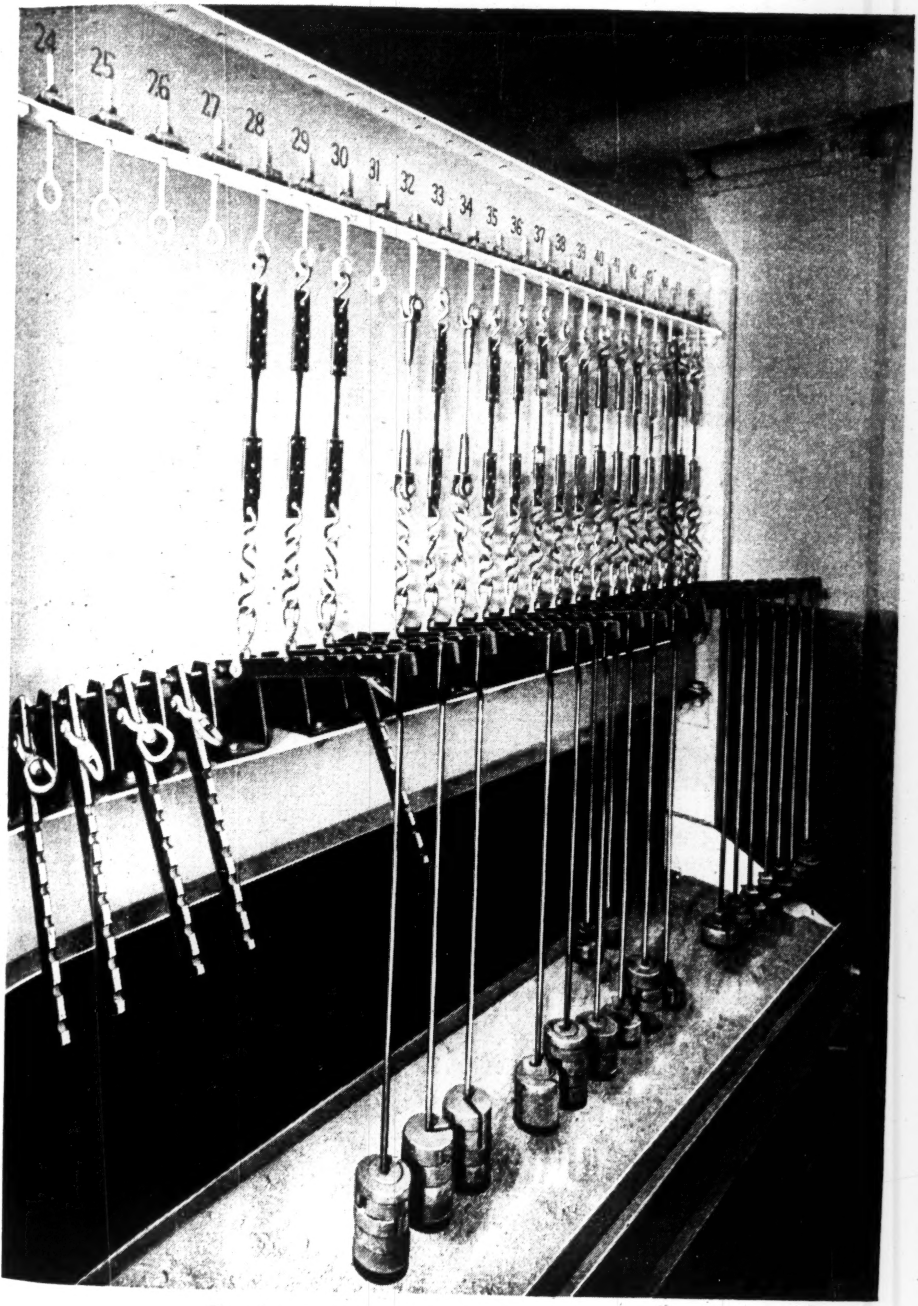
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FIG. 4.—Creep Rack Showing Lever System.

TABLE III.—TYPICAL CREEP DATA—LONGITUDINAL TEST SPECIMENS CUT FROM LEAD CABLE SLEEVING.

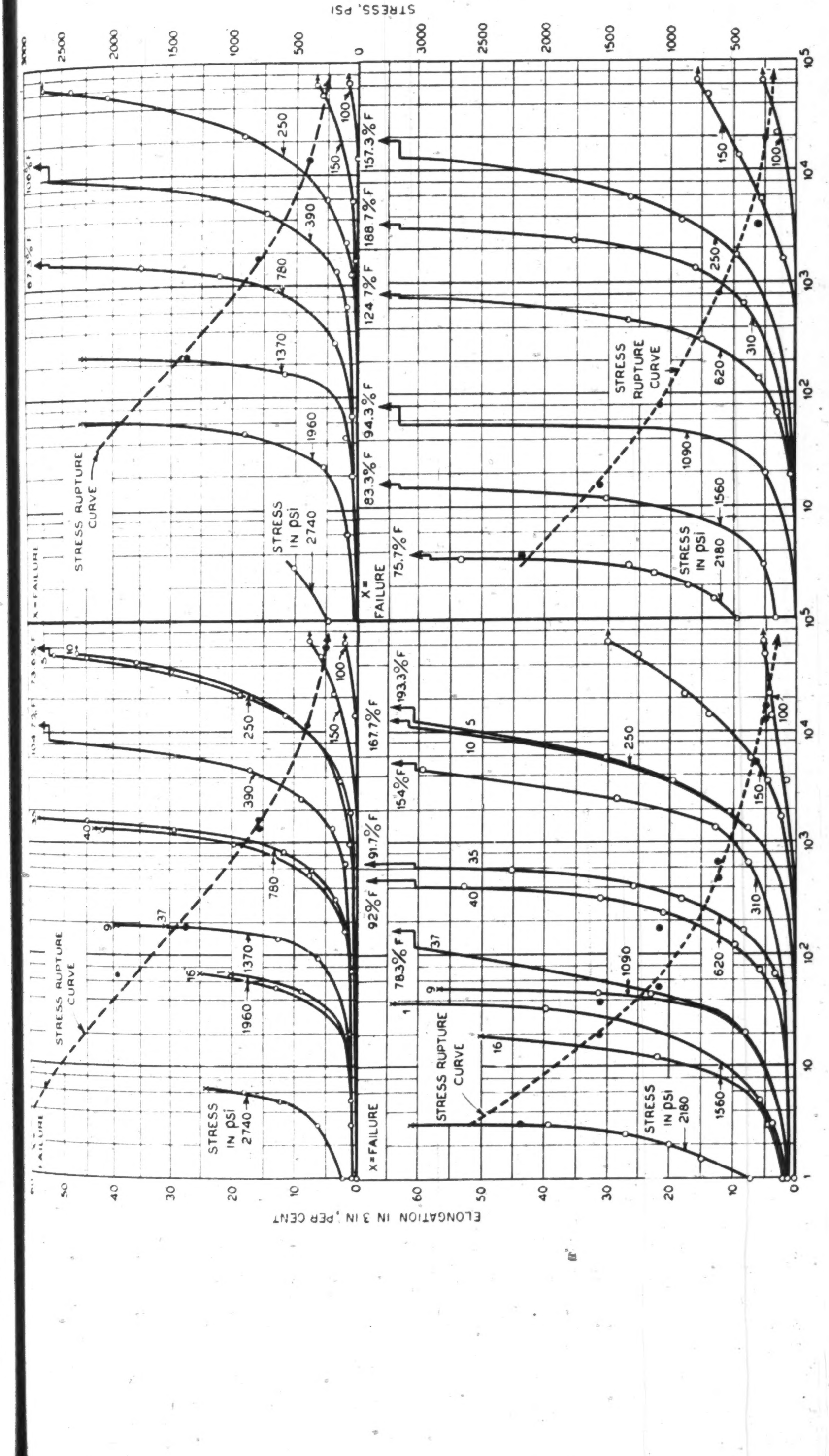
Sleeve No. 6, Chemical Lead (Manufacturer A)

Specimen No. 25		Specimen No. 27		Specimen No. 19		Specimen No. 18		Specimen No. 21		Specimen No. 22		Specimen No. 23	
Initial Stress—70 Per Cent of Ultimate Tensile Strength (1580 psi.)		Initial Stress—50 Per Cent of Ultimate Tensile Strength (1130 psi.)		Initial Stress—35 Per Cent of Ultimate Tensile Strength (790 psi.)		Initial Stress—20 Per Cent of Ultimate Tensile Strength (450 psi.)		Initial Stress—250 psi.		Initial Stress—150 psi.		Initial Stress—100 psi.	
Time, hr.	Elongation in 3 in., per cent	Time, hr.	Elongation in 3 in., per cent	Time, hr.	Elongation in 3 in., per cent	Time, hr.	Elongation in 3 in., per cent	Time, hr.	Elongation in 3 in., per cent	Time, hr.	Elongation in 3 in., per cent	Time, hr.	Elongation in 3 in., per cent
0.08	2.00	6	2.00	20	0.30	19	0.30	1 250	0	1 660	0	1 610	0
0.17	2.70	24	5.3	44	0.70	67	0.30	1 800	0	3 600	0	3 680	0
0.25	3.30	48	8.7	116	1.70	139	0.30	2 500	0	5 710	0	5 690	0
0.50	4.70	72	11.3	140	2.00	211	0.30	3 650	0	13 970	0	13 900	0
0.75	5.7	96	14.7	164	2.00	307	0.30	5 810	0	21 860	0.10	21 800	0
1.00	6.0	168	25.7	188	2.30	475	0.30	14 110	0.47	48 240	0.20	48 190	0
1.25	7.0	192	33.3	212	2.70	739	0.30	21 960	1.20	64 680	0.27 →	63 070	0
1.5	8.0	201	47.0 f	284	3.70	907	0.70	48 340	1.80			64 630	0 →
2.0	9.3			308	3.70	1 080	0.70	55 630	1.80				
3.0	11.7			332	4.00	1 240	0.70	63 890	1.90 →				
4.0	14.0			356	4.70	1 510	0.70						
5.0	17.7			381	4.70	1 900	1.00						
6.5	23.3			549	6.7	3 380	1.30						
14	41.7 f			621	7.0	5 280	2.00						
				789	9.3	8 950	3.30						
				957	11.0	13 320	5.3						
				1120	13.3	15 480	6.0						
				1220	14.3	47 900	22.0						
				1320	15.7	48 720	23.3						
				1480	18.3	49 420	24.7						
				1890	27.3	55 090	24.8						
				2130	38.7	55 250	27.0 f						
				2170	48.0 f								

f denotes total elongation after fracture of test specimen. → denotes no failure.

TABLE IV.—RUPTURE STRESS FOR TIME INDICATED.

Material	Designation	Direction of Extrusion	Rupture Stress, psi. 1 hr.	5 hr.	10 hr.	100 hr.	1000 hr.	10,000 hr.	50,000 hr.	60,000 hr.	70,000 hr.
SLEEVES											
Lead-0.9% Sb	Nos. 1 and 10	Transverse	....	2760	2495	1620	875	395	260	250	...
		Longitudinal	....	....	2745	1730	955	440	280	275	...
Lead-0.7% Sb	Nos. 3 and 9	Transverse	....	2265	1945	1055	590	300	175	170	...
		Longitudinal	....	2050	1820	1135	630	305	210	205	...
Lead-3% Sn	Nos. 4 and 5	Transverse	....	2380	2180	1545	960	520	280	265	...
		Longitudinal	....	2200	2035	1475	960	525	285	275	...
Secondary lead	Nos. 2 and 7	Transverse	1700	1440	1335	970	645	400	235	220	...
		Longitudinal	1845	1540	1420	1030	710	405	245	230	...
Detinned lead	Nos. 13 and 14	Transverse	1725	1520	1425	1090	765	470	300	290	...
		Longitudinal	1750	1510	1405	1085	775	520	385	372	...
Chemical lead	Nos. 6 and 8	Transverse	....	1705	1595	1235	890	600	450	440	
		Longitudinal	....	1865	1720	1270	900	610	450	430	
Chemical lead	Nos. 11 and 12	Transverse	1785	1590	1495	1120	775	525	420	415	
		Longitudinal	1735	1560	1425	1145	798	550	438	430	
TAPE											
High-purity, fire-refined lead	No. 68D	Longitudinal	1260	1100	1030	820	630	445	340	330	325
Chemical lead	No. 89F	Longitudinal	1480	1295	1220	990	780	610	535	525	520
High-purity fire-refined lead-0.06% Te	No. 60A	Longitudinal	....	1695	1570	1140	745	420	240	225	215
Chemical lead-0.07% Te	No. 92F	Longitudinal	....	....	1855	1370	940	620	475	465	450
Chemical lead-0.29% Sb	No. 84A	Longitudinal	....	1520	1415	1080	760	485	340	325	315
Chemical lead-1.0% Sb	No. 91F	Longitudinal	....	....	....	1565	1095	660	395	370	360
High-purity, fire-refined lead-0.02% Ca	No. 29E	Longitudinal	....	2630	2475	1975	1490	1030	975	725	690
High-purity, fire-refined lead-0.027% Ca	No. 63C	Longitudinal	....	....	2450	2030	1625	1250	1030	1010	985
Chemical lead-0.013% Ca	No. 76L	Longitudinal	....	1580	1480	1160	855	580	435	425	415
Chemical lead-0.024% Ca	No. 77L	Longitudinal	....	2175	2050	1645	1250	900	695	675	660
Chemical lead-0.033% Ca	No. 74H	Longitudinal	....	....	2695	2165	1570	1265	1050	1030	1020
Chemical lead-0.036% Ca	No. 54C	Longitudinal	....	....	3170	2585	1960	1355	975	930	900
Chemical lead-0.043% Ca	No. 55A	Longitudinal	....	....	3120	2530	1940	1355	940	890	855
Chemical lead-0.05% Ca	**No. 10B**	**Longitudinal**	**....**	**4050**	**3780**	**2900**	**2080**	**1430**	**1100**	**1080**	**1050**

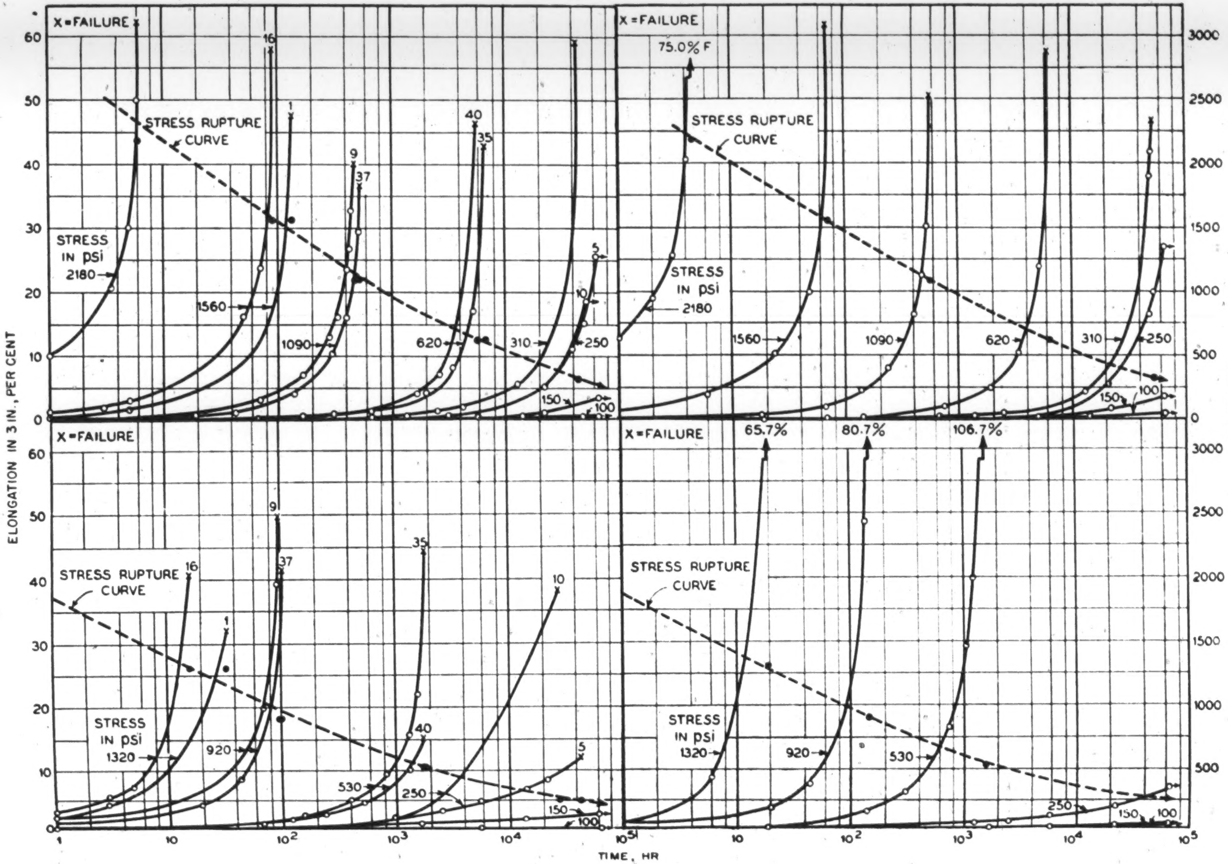


Transverse Test

Longitudinal Test

FIG. 5.—Creep of Specimens from Lead Antimony Sleeves.

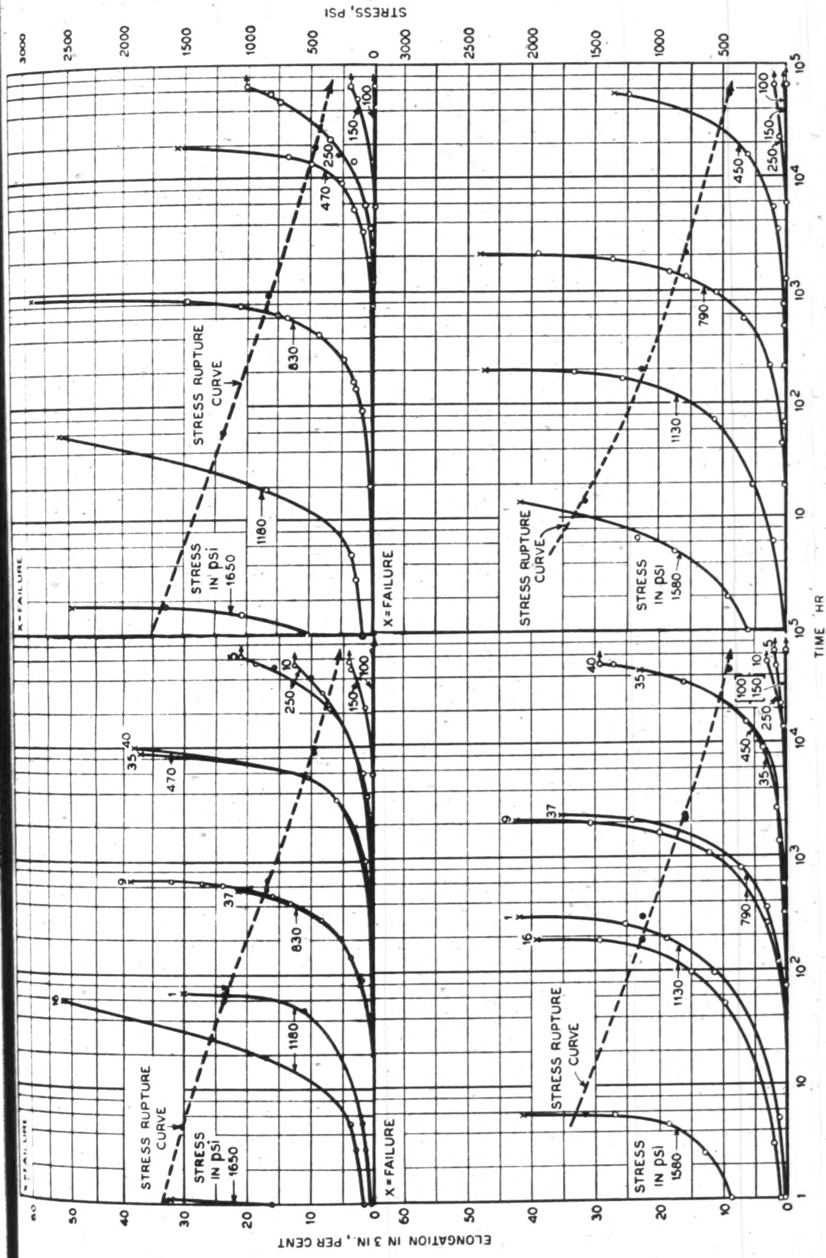
Upper Curves—Sleeve No. 1—Chemical Lead-0.9 per cent Antimony
Lower Curves—Sleeve No. 3—Secondary Lead-0.7 per cent Antimony



Transverse Test

Longitudinal Test

FIG. 6.—Creep of Specimens from Lead and Lead Alloy Sleeves.

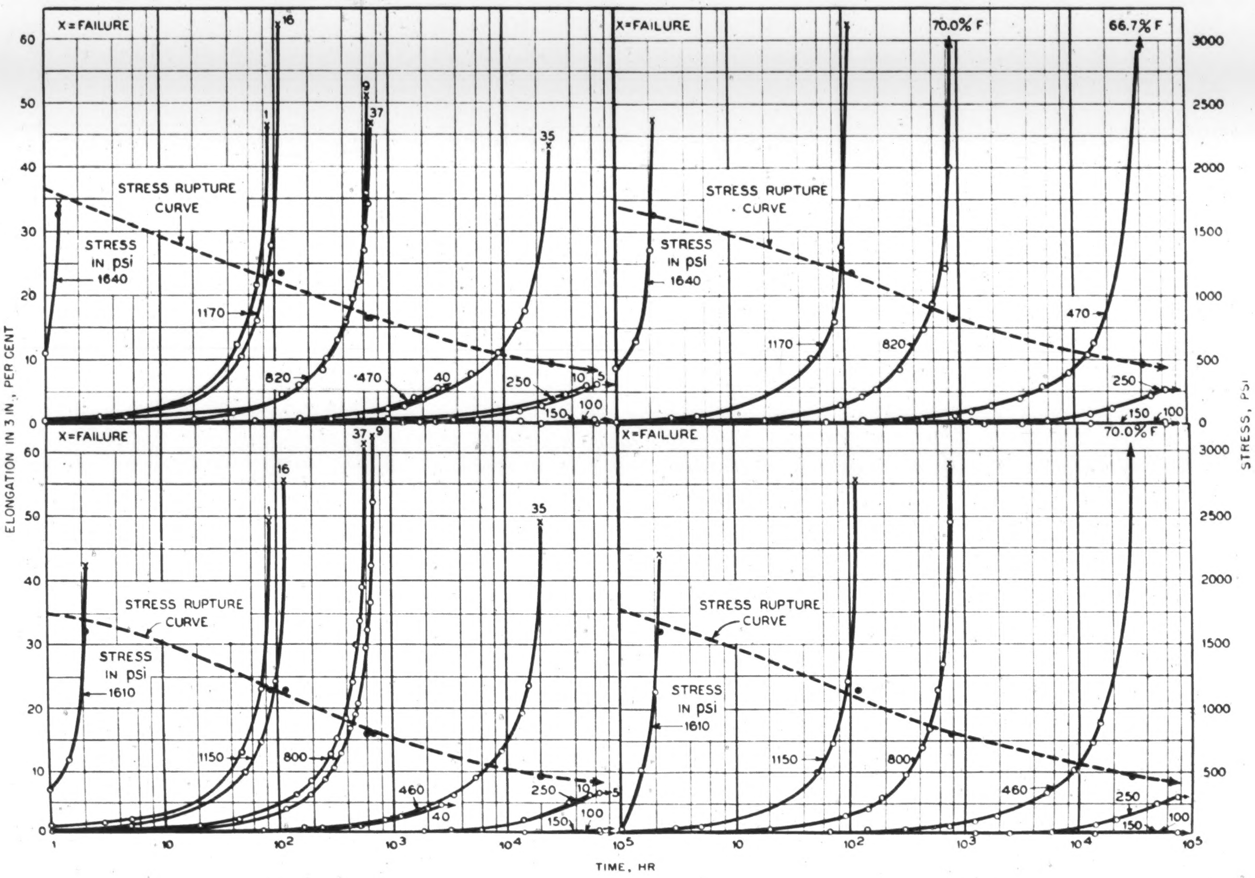


Transverse Test

Longitudinal Test

FIG. 7.—Creep of Specimens from Lead Sleeves.

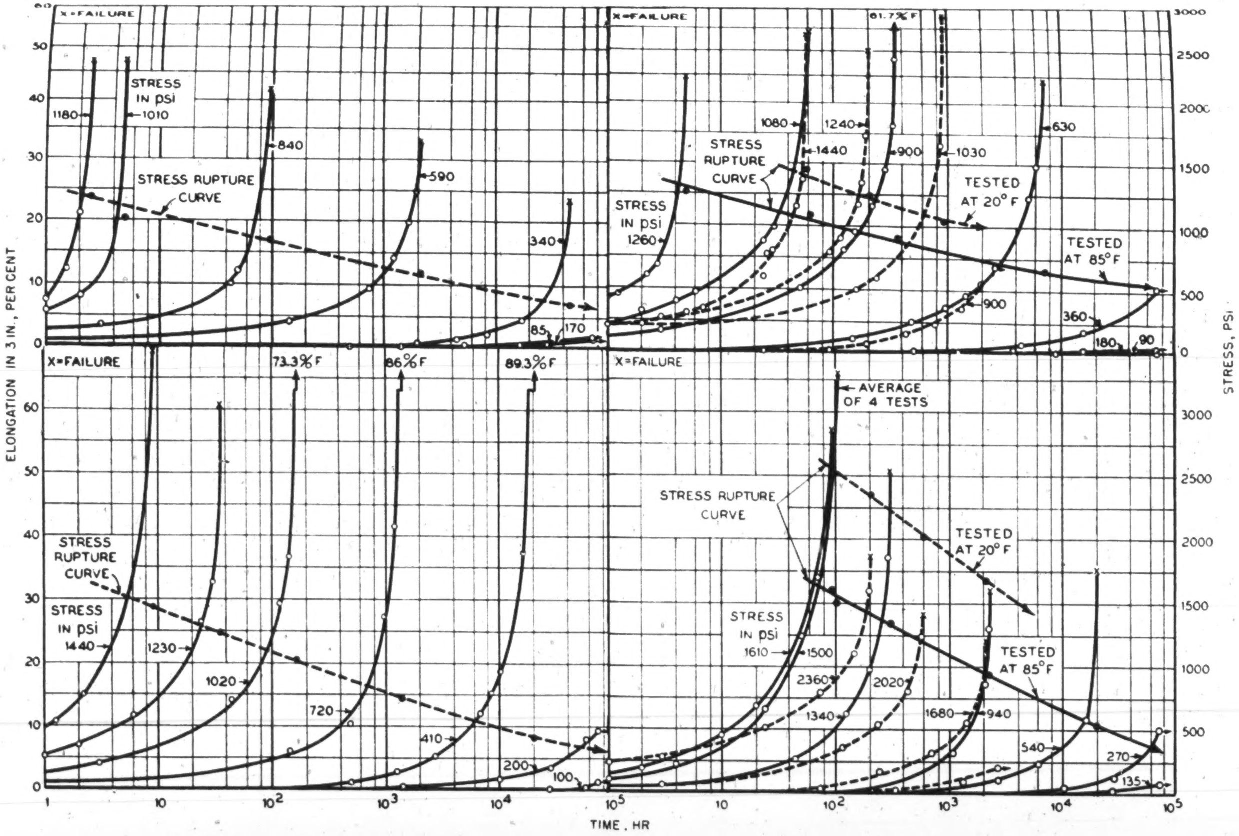
Upper Curves—Sleeve No. 13—Detinned Lead
Lower Curves—Sleeve No. 6—Chemical Lead (Manufacturer A)



Transverse Test Longitudinal Test

FIG. 8.—Creep of Specimens from Chemical Lead (Manufacturer B).

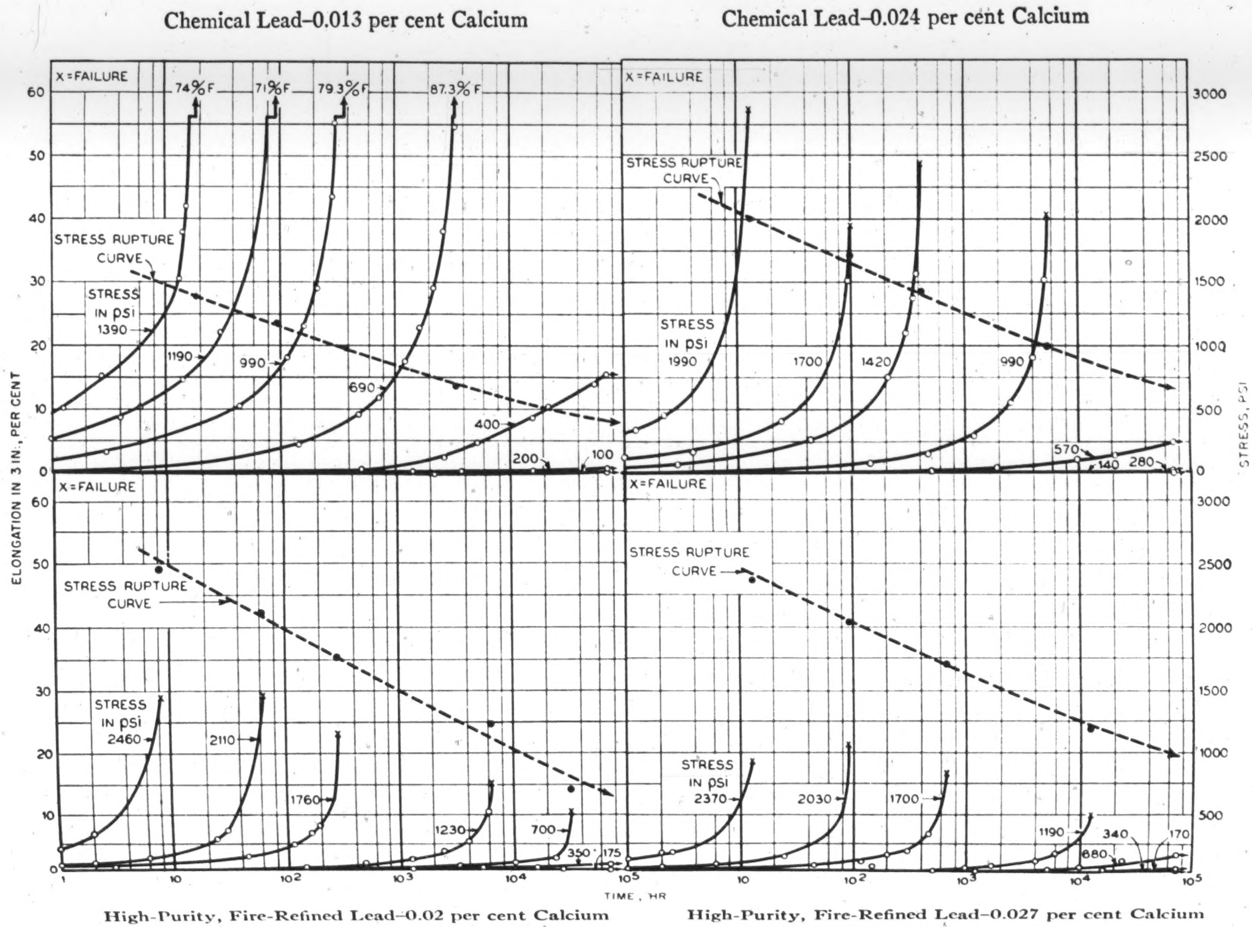
Upper Curves—Sleeve No. 11
Lower Curves—Sleeve No. 12



Chemical Lead-0.29 per cent Antimony

Chemical Lead-1 per cent Antimony

FIG. 9.—Creep of Specimens from Experimental Lead and Lead-Alloy Tapes.

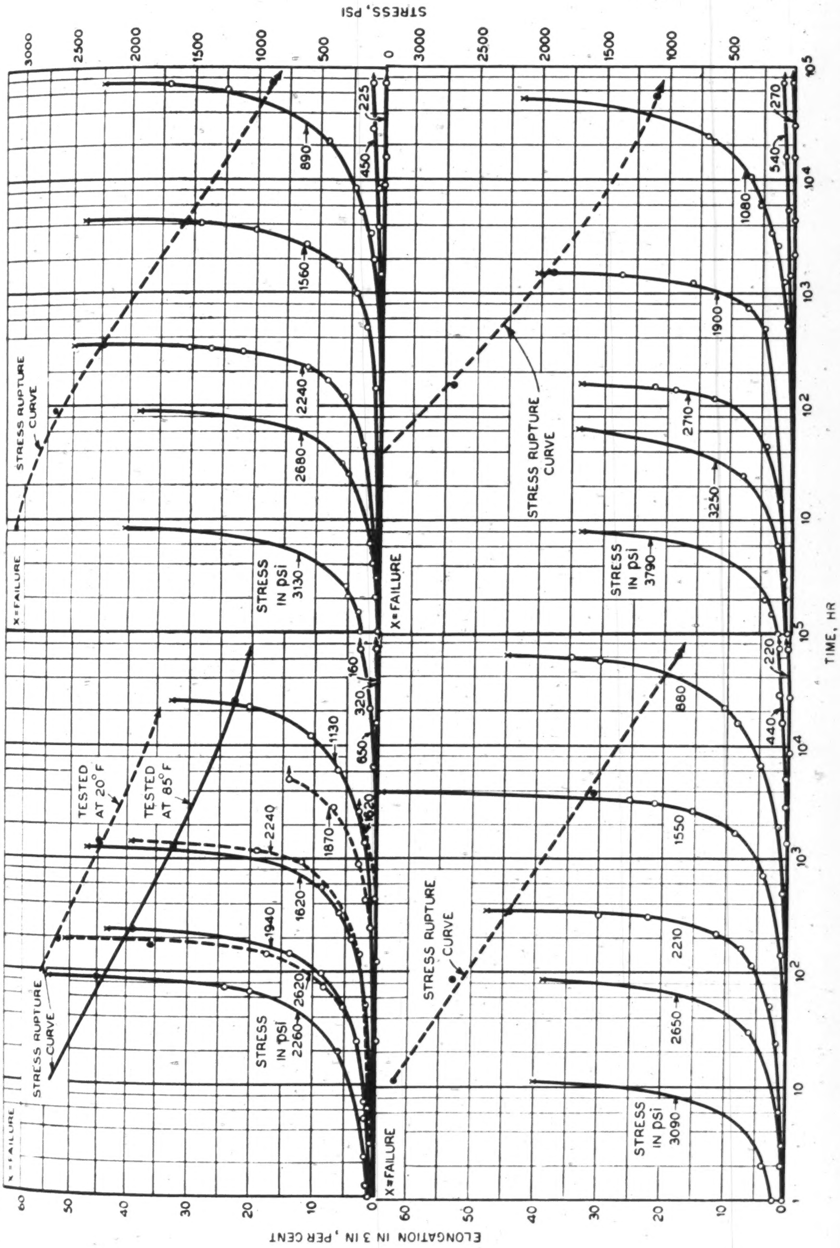

Chemical Lead–0.013 per cent Calcium

Chemical Lead–0.024 per cent Calcium

High-Purity, Fire-Refined Lead–0.02 per cent Calcium

High-Purity, Fire-Refined Lead–0.027 per cent Calcium

FIG. 10.—Creep of Specimens from Experimental Lead-Calcium Tapes.



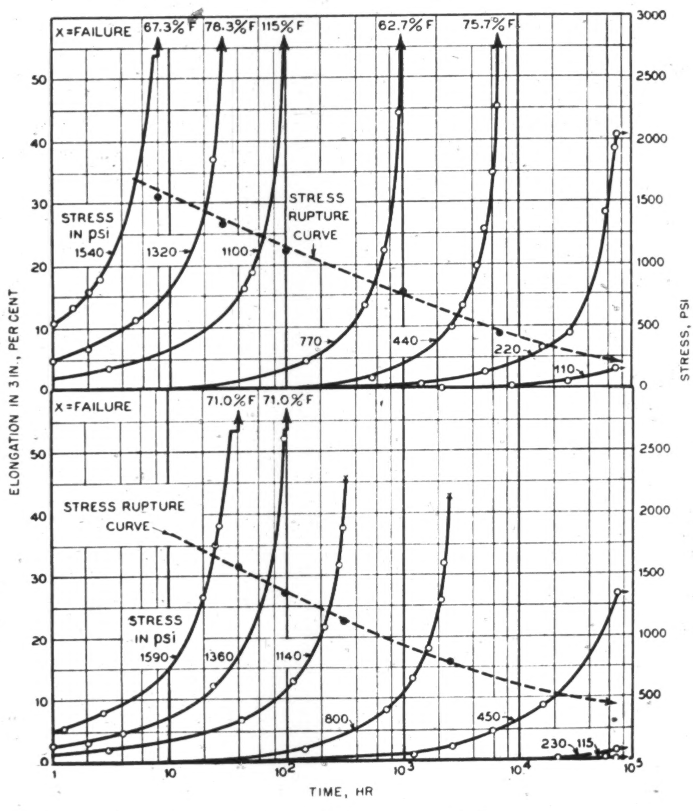
Chemical Lead-0.05 per cent Calcium

Chemical Lead-0.043 per cent Calcium

FIG. 11.—Creep of Specimens from Experimental Lead-Calcium Tapes.

stresses, cathetometer readings (Fig. 3) were taken with a precision of ± 0.07 per cent. The elongation-time measurements were made, until failure occurred, on a series of specimens from each sleeve and tape, after which the total elongation across the break was determined. Measurements are still being recorded on specimens which have not yet failed.

High-Purity, Fire-Refined Lead–0.06 per cent Tellurium



Chemical Lead–0.07 per cent Tellurium

FIG. 12.—Creep of Specimens from Experimental Lead-Tellurium Tapes.

Table III shows typical elongation-time data for one of the alloys tested. For better presentation, these values have been replotted as shown in Fig. 7. The one- and two-digit numbers on the curves identify individual specimens; the three- and four-digit numbers, the test stresses. On curves involving no duplicate specimens, the identifying numbers have been omitted. Similar curves for the other alloys studied in this investigation are shown in Figs. 5 to 12, inclusive. The stress-rupture data, shown on these eight figures as limiting parameters, are summarized in Table IV.

Photomicrographs of sleeve samples selected to show typical changes in structure which occurred during prolonged loading, and to show the nature of the creep failure, are included in Figs. 19 to 24.

DISCUSSION OF DATA

Sleeving:

A study of the data in Table II shows no significant difference in the tensile strength or elongation of duplicate sleeves, of check sleeves as compared to the original sleeves, or between transverse and longitudinal specimens of the same alloy. In the case of the tensile strengths, the greatest spread was less than 6 per cent. All of the alloy sleeves were stronger than the unalloyed lead sleeves in the short-time tension tests. It is also evident from these data that there was no significant change in tensile strength or elongation as a result of aging at room temperature for 8 yr.

A comparison of the tensile data in Table II with the stress-rupture data in Table IV shows that, while the stress-rupture data can be correlated with the tensile data at high stresses, at low stresses there is no correlation.

While stress-rupture data alone, such as those shown in Fig. 13 for the sleeving alloys, have been used by many investigators to evaluate the creep characteristics of materials, such data can be applied in designs only when unlimited deformation short of failure is permissible. In many applications, no deformation can be tolerated, in others such as lead cable sleeves only a limited deformation is permissible. Therefore, to make the creep data more useful, a derived stress-time relationship showing the stress required to produce a fixed elongation is frequently used. Such data for stresses ranging from 100 to 1500 psi. are given in Table V. The stress values required to produce 3 per cent elongation have also been plotted in Fig. 14. Similar derived data are shown in Table VI and in Figs. 15 to 18 for the tape alloys.

Space does not permit the presentation of all of the creep data on the duplicate or check sleeves. This omission is justified because, as illustrated by the four curves shown in Fig. 8 for the two Chemical-lead sleeves supplied by manufacturer B, there was no significant difference between the test results on different sleeves of the same alloy. The results of all the stress-rupture tests have, however, been included in the data given in Table IV and were used in computing the average values and the ratings for the various alloys shown in Tables V and VI.

Analysis of the creep data shows that, except for the secondary-lead sleeves, there was very little difference in the creep resistance (resistance to deformation under continuously applied load) of transverse and longitudinal specimens of the same alloy. However, as noted by Moore and his co-workers (**1, 2**), there was a tendency for the rupture stress to be slightly higher at low stresses for the longitudinal specimens. In nearly all cases the longitudinal specimens showed greater elongation at failure than did the transverse specimens. This was probably due, in part, to the striated structure frequently observed in extruded material; in part to the tendency of small inclusions such as oxides and foreign material to segregate in longitudinal planes thereby causing the formation of voids in lead samples subjected to prolonged transverse tensile loads. Variations in alloy composition resulting from the extrusion of cored ingots may also be partially responsible for this difference.

The lead-antimony alloys which failed at low stresses had very high elongations. This was probably due to their known tendencies to recrystallize after deformation, thus minimizing the building up of high local stresses sufficient to cause grain-boundary separation and consequent brittle fractures.

Further study of the data in Table V

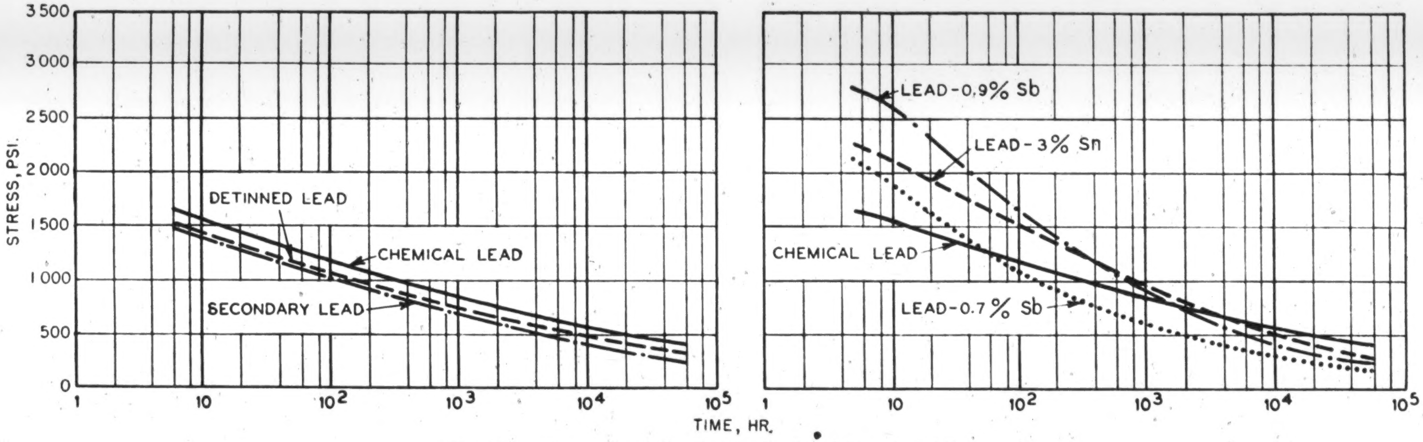


FIG. 13.—Stress-Rupture Curves for Lead Sleeving Alloys.

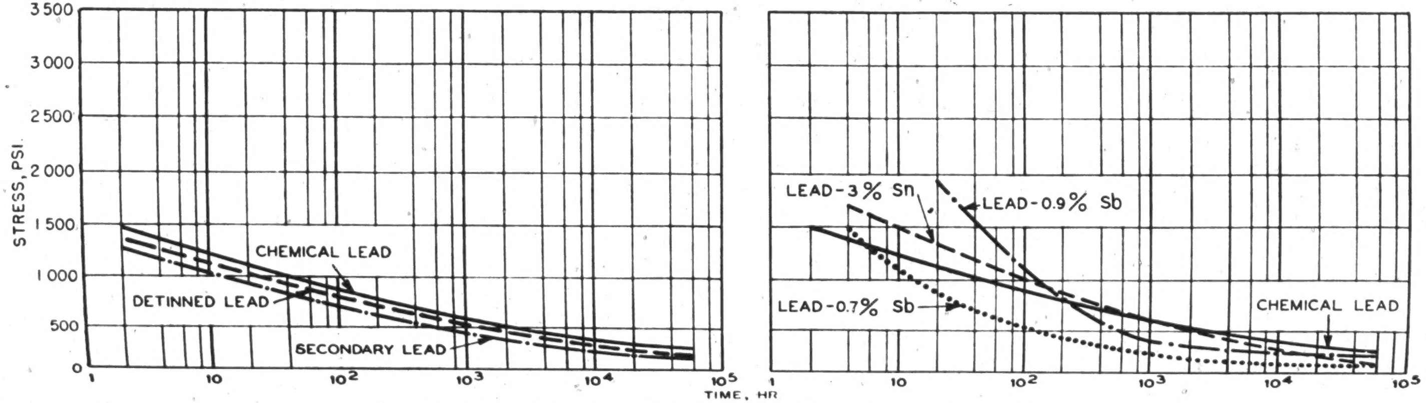


FIG. 14.—Creep Resistance of Lead Sleeving Alloys at 3 per cent Elongation.
NOTE.—Horizontal scales in Figs. 13 and 14 are the time required to produce 3 per cent elongation.

FIG. 14.—Creep Resistance of Lead Sleeving Alloys at 3 per cent Elongation.

NOTE.—Horizontal scales in Figs. 13 and 14 are the time required to produce 3 per cent elongation.

TABLE V. AVERAGE CREEP RESISTANCE OF SPECIMENS CUT FROM LEAD SLEEVING MATERIALS.*

Material	Sleeve	Manufacturer	1500 psi.	1250 psi.	1000 psi.	750 psi.	500 psi.	250 psi.	150 psi.	100 psi.
TIME IN HOURS REQUIRED TO PRODUCE FAILURE AT INDICATED STRESS										
Lead-0.9% Sb	Nos. 1 and 10	A	161 (1)	335 (1)	755 (2)	1885 (3)	6 150 (5)	>65 000		
Lead-0.7% Sb	Nos. 3 and 9	B	28.5 (3)	61.5 (4)	153.5 (6)	478 (7)	1 975 (7)	20 750 (7)		
Lead-3% Sn	Nos. 4 and 5	B	103.5 (2)	285 (2)	860 (1)	2875 (2)	11 350 (3)	>65 000		
Secondary lead	Nos. 2 and 7	B	5.2 (7)	21.4 (7)	104.5 (7)	592 (6)	4 000 (6)	46 000 (6)		
Detinned lead	Nos. 13 and 14	B	5.6 (6)	33.3 (6)	180 (5)	1175 (5)	9 950 (4)	>65 000		
Chemical lead	Nos. 6 and 8	A	24.8 (4)	102.5 (3)	525 (3)	2950 (1)	27 500 (1)	>65 000		
Chemical lead	Nos. 11 and 12	B	7.9 (5)	42.8 (5)	255 (4)	1365 (4)	15 750 (2)	>65 000		
TIME IN HOURS REQUIRED TO PRODUCE 5 PER CENT ELONGATION IN 3 IN. AT INDICATED STRESS										
Lead-0.9% Sb	Nos. 1 and 10	A	67.2 (1)	127 (1)	240 (1)	500 (3)	1 160 (5)	5 610 (6)	40 600 (6)	>65 000
Lead-0.7% Sb	Nos. 3 and 9	B	5.3 (3)	12.6 (3)	34 (6)	95 (7)	250 (7)	1 150 (7)	6 080 (7)	47 000 (7)
Lead-3% Sn	Nos. 4 and 5	B	18.2 (2)	64.5 (2)	230 (2)	834 (1)	3 360 (3)	15 950 (4)	>56 500 (4)	>65 000
Secondary lead	Nos. 2 and 7	B	....	5.0 (4)	22.8 (7)	128 (6)	870 (6)	12 500 (5)	43 100 (5)	>65 000
Detinned lead	Nos. 13 and 14	B	....	....	42.3 (5)	380 (4)	3 620 (2)	17 750 (3)	>65 000	>65 000
Chemical lead	Nos. 6 and 8	A	....	....	91.3 (3)	800 (2)	7 850 (1)	>65 000 (1)	>65 000	>65 000
Chemical lead	Nos. 11 and 12	B	....	....	54.4 (4)	250 (5)	2 500 (4)	48 100 (2)	>65 000	>65 000
TIME IN HOURS REQUIRED TO PRODUCE 3 PER CENT ELONGATION IN 3 IN. AT INDICATED STRESS										
Lead-0.9% Sb	Nos. 1 and 10	A	45.7 (1)	81.8 (1)	142 (1)	260 (3)	600 (5)	3 440 (6)	19 500 (6)	>65 000
Lead-0.7% Sb	Nos. 3 and 9	B	<2 (3)	6.5 (3)	18.8 (6)	50.2 (7)	139 (7)	710 (7)	3 925 (7)	19 400 (7)
Lead-3% Sn	Nos. 4 and 5	B	9 (2)	35.7 (2)	138 (2)	520 (1)	2 060 (3)	11 500 (3)	46 500 (4)	>65 000
Secondary lead	Nos. 2 and 7	B	....	2.7 (4)	11.7 (7)	66.3 (6)	440 (6)	5 010 (5)	>37 700 (5)	>61 500 (6)
Detinned lead	Nos. 13 and 14	B	....	....	21.4 (5)	225 (4)	2 180 (2)	10 600 (4)	49 750 (3)	>65 000
Chemical lead	Nos. 6 and 8	A	....	....	55.3 (3)	485 (2)	4 010 (1)	>59 000 (1)	>65 000	>65 000
Chemical lead	Nos. 11 and 12	B	....	....	31.2 (4)	145 (5)	1 270 (4)	24 250 (2)	>65 000	>65 000
TIME IN HOURS REQUIRED TO PRODUCE 1 PER CENT ELONGATION IN 3 IN. AT INDICATED STRESS										
Lead-0.9% Sb	Nos. 1 and 10	A	7.8 (1)	17.6 (1)	32.0 (2)	63.2 (4)	138 (5)	1 170 (5)	6 450 (6)	38 000 (6)
Lead-0.7% Sb	Nos. 3 and 9	B	<1 (3)	<1 (3)	<4.8 (5)	<12.8 (6)	39.5 (7)	228 (7)	1 525 (7)	5 500 (7)
Lead-3% Sn	Nos. 4 and 5	B	<1.8 (2)	7.3 (2)	39.6 (1)	148 (1)	605 (3)	4 640 (3)	14 100 (5)	>65 000
Secondary lead	Nos. 2 and 7	B	....	....	....	12.1 (7)	120 (6)	710 (6)	>18 100 (3)	>37 000 (5)
Detinned lead	Nos. 13 and 14	B	....	....	2.7 (6)	64 (3)	740 (2)	4 410 (4)	18 600 (4)	>65 000
Chemical lead	Nos. 6 and 8	A	....	....	12.5 (3)	142 (2)	1 220 (1)	25 500 (1)	>65 000	>65 000
Chemical lead	Nos. 11 and 12	B	....	....	6.9 (4)	33.7 (5)	285 (4)	8 575 (2)	>65 000	>65 000
ELONGATION IN 3 IN. FOR 60,000 HR. AT INDICATED STRESS, PER CENT^{b}										
Lead-0.9% Sb	Nos. 1 and 10	A						52.1^{a}	7.68 (6)	1.55 (5)
Lead-0.7% Sb	Nos. 3 and 9	B						14.5^{b}	21.29 (7)	5.74 (7)
Lead-3% Sn	Nos. 4 and 5	B						42.6^{c}	5.02 (4)	1.10 (4)
Secondary lead	Nos. 2 and 7	B						6.5^{e}	5.68 (5)	1.70 (6)
Detinned lead	Nos. 13 and 14	B						6.6^{d} (3)	3.58 (3)	0.37 (3)
Chemical lead	Nos. 6 and 8	A						2.1^{d} (1)	0.08 (1)	0.00 (1)
Chemical lead	Nos. 11 and 12	B						6.0^{d} (2)	0.28 (2)	0.00 (1)

* Figures in parentheses represent rating of sleeving alloys.

[a] For 3 out of 6 specimens. Remaining 3 failed in 44 000 to 57 000 hr.

[b] 5 out of 6 specimens failed in 13 000 to 29 000 hr.

[c] For 5 out of 6 specimens; 1 specimen failed in 60 000 hr.

[d] No specimens failed.

[e] 4 out of 6 specimens failed in 26 000 to 49 000 hr. Measurements on 1 specimen discontinued after 22 000 hr.

TABLE VI.—CREEP RESISTANCE OF SPECIMENS CUT FROM LABORATORY EXTRUDED LEAD-ALLOY TAPES.[a]

Material	Tape	3000 psi.	2500 psi.	2000 psi.	1500 psi.	1250 psi.	1000 psi.	750 psi.	500 psi.	400 psi.	250 psi.
TIME IN HOURS REQUIRED TO PRODUCE FAILURE AT INDICATED STRESS											
High-purity, fire-refined lead	No. 68D	....	....	....	....	1.1 (14)	14 (14)	235 (14)	4 700 (14)	19 500 (13)	>70 000
Chemical lead	No. 89F	....	....	....	....	7.5 (13)	92 (13)	1 350 (11)	>70 000	>70 000	>70 000
High-purity, fire-refined lead-0.06% Te.	No. 60A	....	....	....	14.3 (10)	55 (10)	220 (11)	950 (13)	5 000 (13)	11 000 (14)	>70 000
Chemical lead-0.07% Te	No. 92F	....	....	....	53 (9)	180 (9)	700 (9)	3 500 (9)	33 500 (9)	>70 000	44 500 (14)
Chemical lead-0.29% Sb	No. 84A	....	....	....	5.6 (12)	30 (12)	175 (12)	1 080 (12)	8 500 (12)	25 500 (12)	>70 000
Chemical lead-1.0% Sb	No. 91F	....	....	....	132 (8)	450 (8)	1 550 (8)	5 400 (8)	25 000 (10)	49 000 (11)	>70 000
High-purity, fire-refined lead-0.02% Ca.	No. 29E	....	8.6 (5)	85 (6)	920 (6)	3 100 (6)	11 000 (6)	41 500 (6)	>70 000	>70 000	>70 000
High-purity, fire-refined lead-0.027% Ca	No. 63C	....	7.7 (6)	118 (5)	2080 (5)	9 800 (5)	61 000 (3)	>70 000	>70 000	>70 000	>70 000
Chemical lead-0.013% Ca	No. 76L	....	....		9 (11)	53 (11)	325 (10)	2 250 (10)	23 500 (11)	>70 000	>70 000
Chemical lead-0.024% Ca	No. 77L		....	13.2 (7)	230 (7)	1 000 (7)	4 900 (7)	32 000 (7)	>70 000	>70 000	>70 000
Chemical lead-0.033% Ca	No. 74H	....	22.7 (4)	222 (4)	2380 (4)	10 700 (4)	>70 000	>70 000	>70 000	>70 000	>70 000
Chemical lead-0.036% Ca	No. 54C	20.7 (2)	136 (2)	850 (2)	5600 (2)	15 000 (2)	44 000 (4)	>70 000	>70 000	>70 000	>70 000
Chemical lead-0.0043% Ca	No. 55A	15.4 (3)	108 (3)	770 (3)	5500 (3)	14 700 (3)	34 000 (5)	>70 000	>70 000	>70 000	>70 000
Chemical lead-0.05% Ca	No. 10B	74 (1)	290 (1)	1300 (1)	6800 (1)	23 000 (1)	>70 000	>70 000	>70 000	>70 000	>70 000
TIME IN HOURS REQUIRED TO PRODUCE 5 PER CENT ELONGATION IN 3 IN. AT INDICATED STRESS											
High-purity, fire-refined lead	No. 68D	<1	<1	<1	<1	<1	<1 (14)	31.5 (13)	1 050 (13)	6 100 (12)	60 000 (11)
Chemical lead	No. 89F	<1	<1	<1	<1	<1	3 (13)	95 (11)	4 100 (10)	18 000 (9)	>70 000
High-purity, fire-refined lead-0.06% Te.	No. 60A	<1	<1	<1	<1	1.9 (10)	17.5 (10)	195 (10)	910 (14)	2 000 (14)	9 100 (14)
Chemical lead-0.07% Te	No. 92F	<1	<1	<1	1.9 (9)	10.4 (9)	74 (9)	600 (9)	4 400 (9)	12 500 (11)	>70 000
Chemical lead-0.29% Sb	No. 84A	<1	<1	<1	<1	<1	5 (12)	11.3 (14)	3 500 (11)	26 500 (8)	34 500 (13)
Chemical lead-1.0% Sb	No. 91F	<1	<1	<1	6 (7)	74 (8)	510 (8)	2 150 (8)	8 400 (8)	17 000 (10)	52 000 (12)
High-purity, fire-refined lead-0.02% Ca.	No. 29E	<1	<1	355 (3)	670 (5)	3 450 (3)	9 200 (4)	24 000 (5)	>70 000	>70 000	>70 000
High-purity, fire-refined lead-0.027% Ca	No. 63C	<1	<1	52 (5)	1250 (3)	5 800 (1)	27 500 (1)	>70 000 (1)	>70 000	>70 000	>70 000
Chemical lead-0.013% Ca	No. 76L	<1	<1	<1	<1	<1	6.4 (11)	82 (12)	1 600 (12)	5 400 (13)	>70 000
Chemical lead-0.024% Ca	No. 77L	<1	<1	<1	2.8 (8)	145 (7)	900 (7)	12 400 (7)	>70 000	>70 000	>70 000
Chemical lead-0.033% Ca	No. 74H	<1	<1	38.5 (6)	520 (6)	2 250 (6)	11 500 (2)	67 000 (3)	>70 000	>70 000	>70 000
Chemical lead-0.036% Ca	No. 54C	2.5 (3)	103 (1)	980 (1)	1400 (2)	3 000 (4)	6 400 (5)	32 000 (4)	>70 000	>70 000	>70 000
Chemical lead-0.043% Ca	No. 55A	4.5 (2)	39 (3)	210 (4)	1140 (4)	2 500 (5)	5 500 (6)	18 000 (6)	>70 000	>70 000	>70 000
Chemical lead-0.05% Ca	No. 10B	26 (1)	90 (2)	375 (2)	1700 (1)	3 650 (2)	11 000 (3)	70 000 (2)	>70 000	>70 000	>70 000
TIME IN HOURS REQUIRED TO PRODUCE 3 PER CENT ELONGATION IN 3 IN. AT INDICATED STRESS											
High-purity, fire-refined lead	No. 68D	<1	<1	<1	<1	<1	<1	7.2 (13)	400 (13)	3 000 (11)	34 000 (12)
Chemical lead	No. 89F	<1	<1	<1	<1	<1	<1	32 (11)	1 850 (10)	8 500 (9)	>70 000
High-purity, fire-refined lead-0.06% Te.	No. 60A	<1	<1	<1	<1	<1	7 (10)	110 (10)	540 (12)	1 130 (14)	4 500 (14)
Chemical lead-0.07% Te	No. 92F	<1	<1	<1	<1	2.9 (9)	27.5 (9)	300 (9)	2 250 (9)	7 000 (10)	67 000 (9)
Chemical lead-0.29% Sb	No. 84A	<1	<1	<1	<1	<1	1.5 (12)	3.6 (14)	250 (14)	1 600 (13)	10 300 (13)
Chemical lead-1.0% Sb	No. 91F	<1	<1	<1	2.5 (8)	36 (8)	310 (8)	1 300 (8)	4 850 (8)	10 300 (8)	35 000 (11)
High-purity, fire-refined lead-0.02% Ca	No. 29E	<1	<1	3.6 (6)	65 (7)	370 (6)	2 100 (6)	6 600 (6)	20 000 (7)	>70 000	>70 000
High-purity, fire-refined lead-0.027% Ca	No. 63C	<1	<1	29 (4)	720 (1)	3 600 (1)	14 000 (1)	50 000 (2)	>70 000	>70 000	>70 000
Chemical lead-0.013% Ca	No. 76L	<1	<1	<1	<1	<1	2.2 (11)	29.5 (12)	700 (11)	2 750 (14)	45 000 (10)
Chemical lead-0.024% Ca	No. 77L	<1	<1	<1	9.4 (6)	60 (7)	420 (7)	5 000 (7)	>70 000	>70 000	>70 000
Chemical lead-0.033% Ca	No. 74H	<1	<1	18 (5)	270 (5)	1 120 (5)	5 800 (3)	36 000 (3)	>70 000	>70 000	>70 000
Chemical lead 0.036% Ca	No. 54C	2 (3)	20.3 (3)	107 (1)	710 (2)	1 430 (3)	2 900 (5)	12 000 (5)	>70 000	>70 000	>70 000
Chemical lead 0.043% Ca	No. 55A	2.6 (2)	21 (2)	105 (2)	580 (3)	2 600 (2)	9 300 (2)	56 000 (1)	>70 000	>70 000	>70 000
Chemical lead 0.05% Ca	No. 10B	13.5 (1)	35 (4)	79 (3)	455 (4)	1 250 (4)	3 000 (4)	19 000 (4)	>70 000	>70 000	>70 000

Time in Hours Required to Produce 1 per cent Elongation in 3 in. at Indicated Stress

High purity, fire refined lead	No. 68D	<1	<1	<1	<1	<1	<1	<1	<1 (14)	450 (13)	11 400 (12)
Chemical lead	No. 89F	<1	<1	<1	<1	<1	<1	5 (11)	375 (10)	1 950 (9)	35 000 (8)
High-purity, fire-refined lead-0.06% Te.	No. 60A	<1	<1	<1	<1	<1	1.7 (10)	40 (10)	220 (11)	440 (14)	1 360 (14)
Chemical lead-0.07% Te	No. 92F	<1	<1	<1	<1	<1	7.7 (9)	78 (8)	430 (9)	1 750 (10)	42 500 (7)
Chemical lead-0.29% Sb	No. 84A	<1	<1	<1	<1	<1	<1	<1	75 (13)	500 (12)	2 700 (13)
Chemical lead-1.0% Sb	No. 91F	<1	<1	<1	<1	3.3 (7)	113 (7)	550 (7)	1 700 (8)	3 800 (8)	13 500 (11)
High-purity, fire-refined lead-0.02% Ca	No. 29E	<1	<1	<1	22 (6)	430 (2)	1 440 (2)	4 350 (3)	7 700 (5)	9 200 (6)	3 300 (9)
High-purity, fire-refined lead-0.027% Ca	No. 63C	<1	<1	7 (3)	175 (2)	1 130 (1)	4 000 (1)	11 800 (1)	43 500 (2)	>70 000	>70 000
Chemical lead-0.013% Ca	No. 76L	<1	<1	<1	<1	<1	<1	3.9 (12)	170 (12)	900 (11)	32 000 (10)
Chemical lead-0.024% Ca	No. 77L	<1	<1	<1	<1	3.2 (8)	9.6 (8)	310 (8)	8 800 (4)	28 000 (3)	>70 000
Chemical lead-0.033% Ca	No. 74H	<1	<1	2 (4)	26 (4)	155 (4)	1 040 (3)	7 000 (2)	45 000 (1)	>70 000	>70 000
Chemical lead-0.036% Ca	No. 54C	<1	3.9 (2)	10.4 (2)	52 (3)	154 (5)	450 (6)	1 080 (6)	2 150 (7)	7 800 (7)	>70 000
Chemical lead-0.043% Ca	No. 55A	<1	4.2 (1)	18 (1)	118 (1)	265 (3)	600 (5)	2 000 (4)	9 200 (3)	19 000 (4)	62 000 (6)
Chemical lead-0.05% Ca	No. 10B	<1	1 (3)	1 (5)	23.5 (5)	140 (6)	640 (4)	1 850 (5)	6 500 (6)	18 000 (5)	>70 000

Elongation in 3 in. at Indicated Stress, per cent

		60,000 hr.			70,000 hr.		
		250 psi.	150 psi.	100 psi.	250 psi.	150 psi.	100 psi.
High-purity, fire-refined lead	No. 68D	(14)	1.4 (10)	1.4 (14)	(14)	1.5 (10)	1.5 (14)
Chemical lead	No. 89F	3.8 (9)	0.0	0.0	4.2 (9)	0.0	0.0
High-purity, fire-refined lead-0.06% Te.	No. 60A	>28 (13)	11.6 (14)	0.4 (12)	47.8 (13)	15.6 (14)	0.0
Chemical lead-0.07% Te	No. 92F	3.10 (8)	0.4 (9)	0.0	3.8 (8)	1.0 (9)	0.05 (9)
Chemical lead-0.29% Sb	No. 84A	11.5 (12)	4.2 (13)	0.5 (13)	11.9 (12)	5.0 (13)	1.0 (13)
Chemical lead-1.0% Sb	No. 91F	6.0 (10)	1.8 (11)	0.0	7.2 (10)	1 9 (11)	0.0
High-purity, fire-refined lead-0.02% Ca.	No. 29E	0.5 (6)	0.0	0.0	0.5 (6)	0.0	0.0
High-purity, fire-refined lead-0.027% Ca	No. 63C	0.2 (3)	0.05 (6)	0.02 (9)	0.3 (2)	0.1 (6)	0.06 (10)
Chemical lead-0.013% Ca	No. 76L	8.9 (11)	3.5 (12)	0.1 (11)	9.8 (11)	4.0 (12)	0.2 (11)
Chemical lead-0.024% Ca	No. 77L	1.2 (7)	0.3 (8)	0.0	1.4 (7)	0.4 (8)	0.0
Chemical lead-0.033% Ca	No. 74H	0.2 (4)	0.08 (7)	0.04 (10)	0.3 (3)	0.2 (7)	0.2 (11)
Chemical lead-0.036% Ca	No. 54C	0.2 (1)	0.0	0.0	0.2 (1)	0.0	0.0
Chemical lead-0.043% Ca	No. 55A	0.4 (5)	0.0	0.0	0.44 (5)	0.0	0.0
Chemical lead-0.05% Ca	No. 10B	0.2 (1)	0.0	0.0	0.35 (4)	0.0	0.0

[a] Figures in parenthesis represent rating of tape alloys.

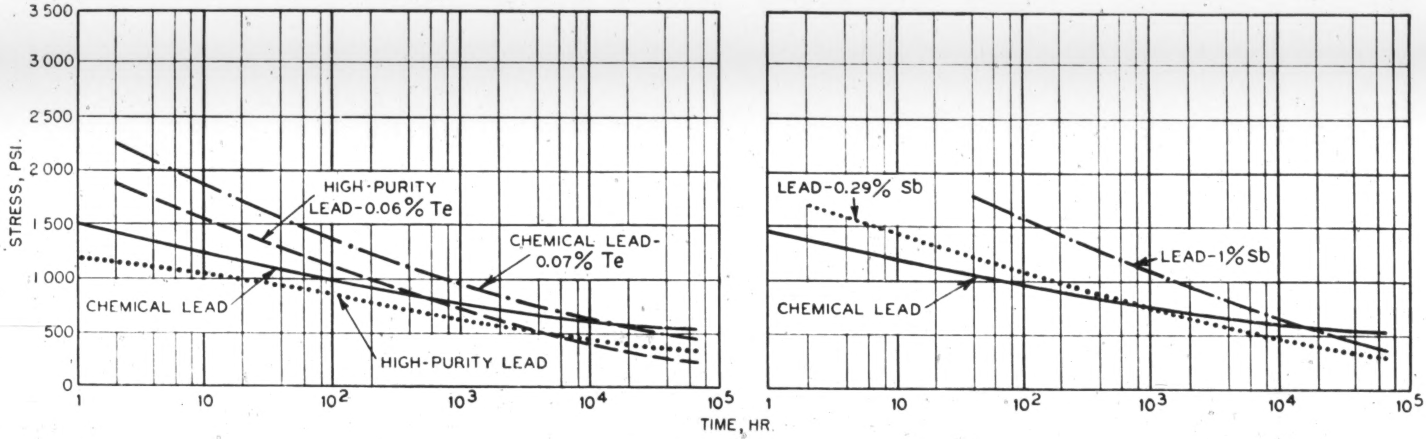


FIG. 15.—Stress-Rupture Curves for Lead Alloy Tapes.

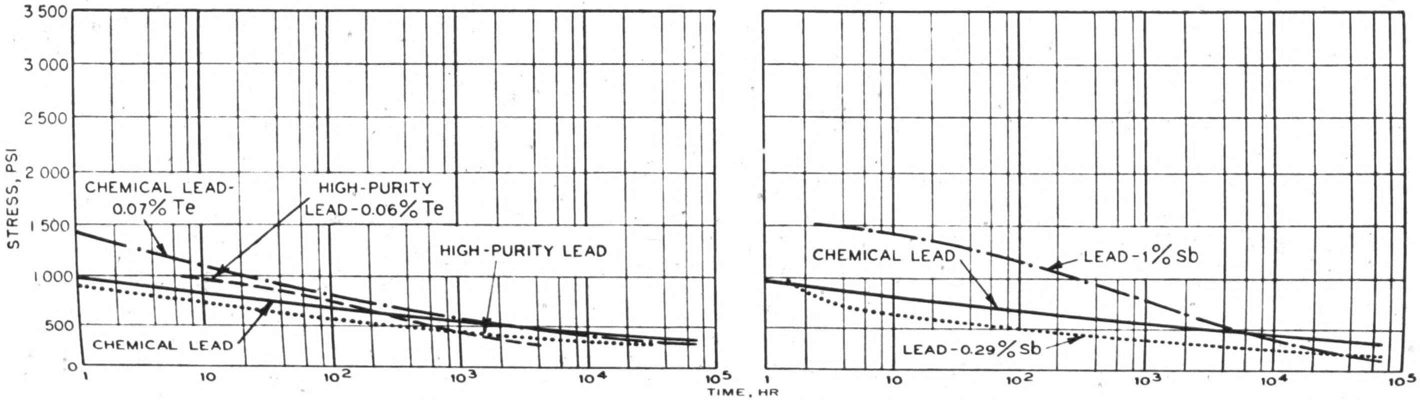


FIG. 16.—Creep Resistance of Lead Alloy Tapes at 3 per cent Elongation.
NOTE.—Horizontal scales in Figs. [illegible] are the time required to produce 3 per cent elongation.

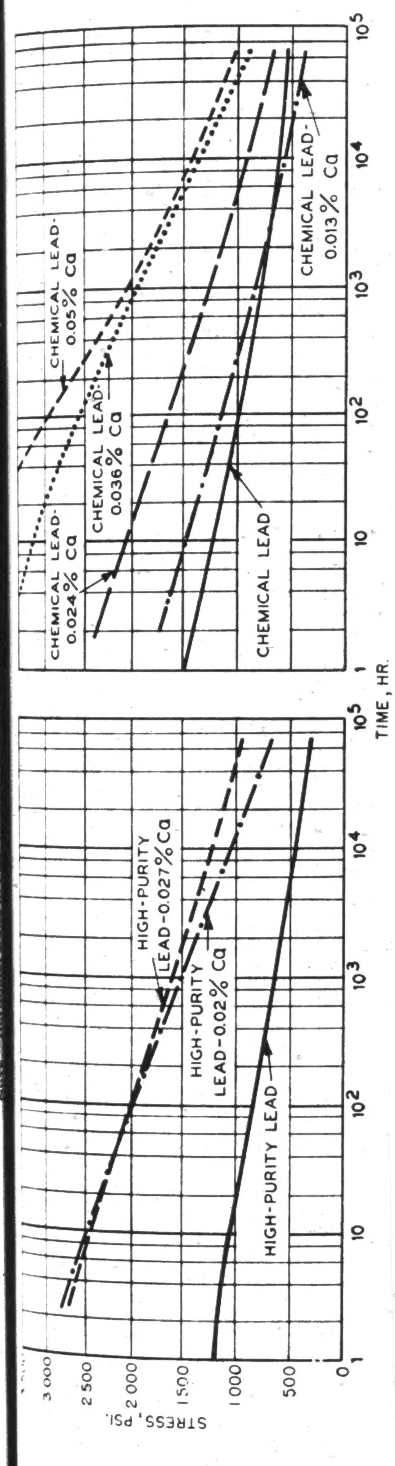


FIG. 17.—Stress-Rupture Curves for Lead Alloy Tapes.

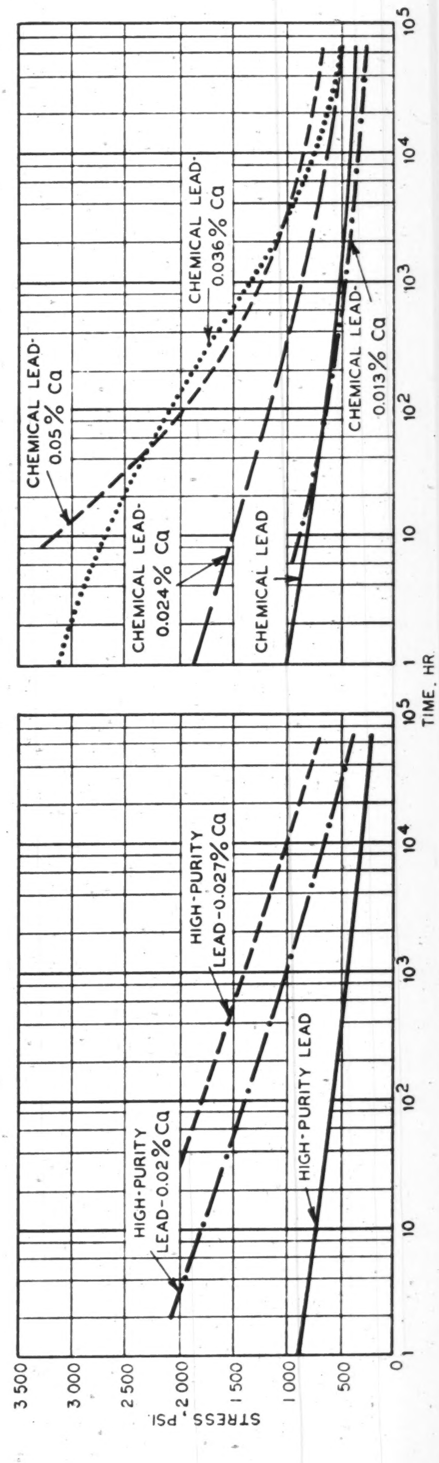


FIG. 18.—Creep Resistance of Lead Alloy Tapes at 3 per cent Elongation.

NOTE.—Horizontal scales in Figs. 17 and 18 are the time required to produce 3 per cent elongation.

shows that none of the specimens from the Chemical-lead sleeves had any measurable elongation at a stress of 100 psi. after 60,000 hr. One of the four specimens from the secondary-lead sleeves likewise showed no measurable elongation under these same conditions. At a stress of 150 psi., the only specimens which showed no measurable creep were some of those taken from the Chemical-lead sleeves, while at a stress of 250 psi. the specimens of all six sleeving alloys had either failed in less than 60,000 hr. or showed measurable elongation. Only the Chemical- and detinned-lead specimens showed no failure at this stress, the Chemical-lead specimens having elongated from 1.7 to 6.9 per cent; those of detinned lead more than 10 but less than 15 per cent in 60,000 hr.

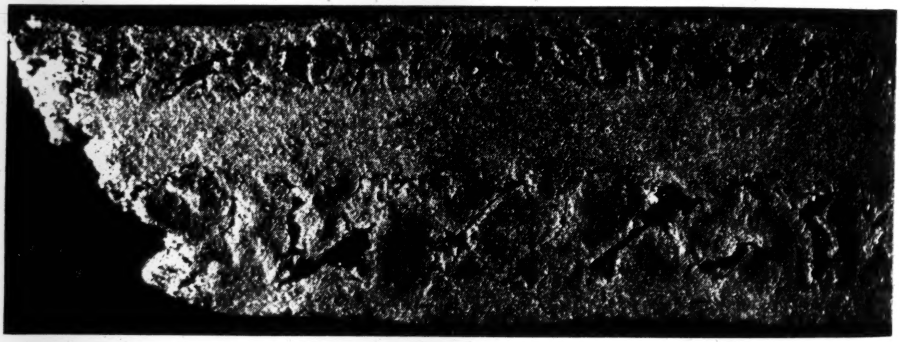
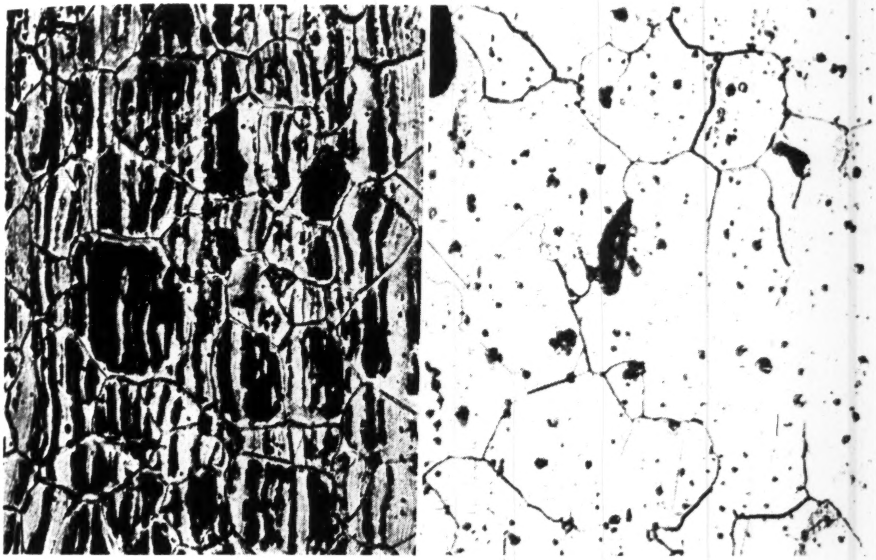


FIG. 19.—Longitudinal Creep Specimen Showing Outer Surface of Secondary Lead-0.07 per cent Antimony Sleeve After Failure in 5950 hr. at 310 psi. (× 5).

The series of photomicrographs in Figs. 19 to 24 illustrate the mechanism of failure and depict the structural changes occurring in some of the sleeving materials during the creep tests. The first six photomicrographs pertain to lead-antimony alloys. Figure 19 shows the surfaces of a lead-0.7 per cent antimony specimen after failure at 310 psi. in 5950 hr. Typical surface cracking and grain-boundary separation are clearly evident. Figure 20 provides a clue which may explain this type of failure. The structure of the unstressed extruded alloy (Fig. 20 (*a*)) consists of large areas (dark in the photomicrograph) of material rich in antimony, and therefore of high tensile strength, surrounded by light areas depleted in antimony near the grain boundaries. These areas are weak and are conducive to failure in this region.

Pronounced structural changes occur when the lead-antimony alloy is stressed for longer periods of time. Recrystallization has taken place and the antimony particles, which cause dispersion hardening, have coalesced to form the dark spots shown in Fig. 20 (*b*). The matrix, containing about 0.2 per cent antimony in solid solution, is relatively weak and recrystallizes readily. These changes probably account for the relatively poor creep resistance of the lead-antimony alloys at low stress where the prolonged loading afforded ample time for recrystallization, whereas at higher stresses failure occurred before recrystallization had time to take place.

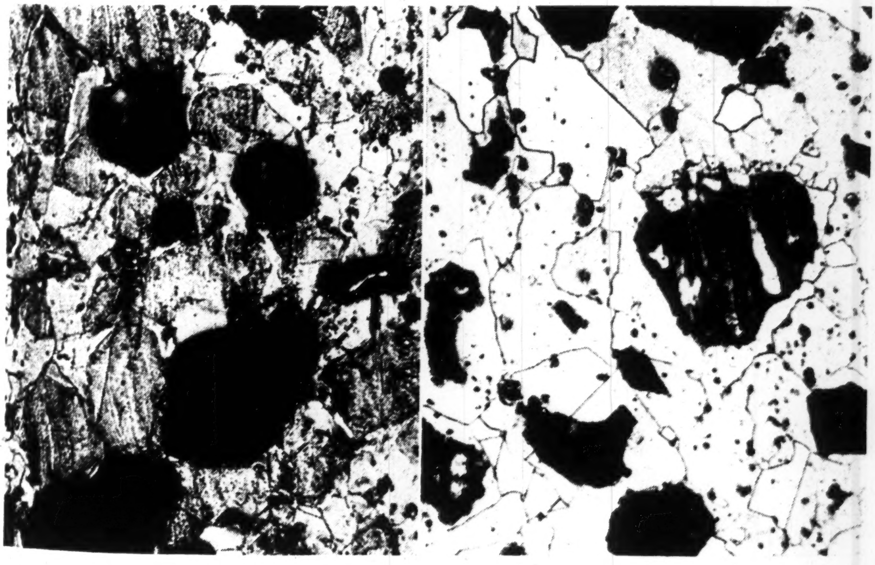
A direct comparison of the structure at failure for a lead-0.9 per cent antimony alloy after short and long periods of stressing is afforded by Fig. 21. After 2460 hr. at 730 psi. there has been only a



(*a*) Unstressed (*b*) After failure in 5950 hr. at 310 psi.

FIG. 20.—Secondary Lead-0.7 per cent Antimony Sleeve (× 250).

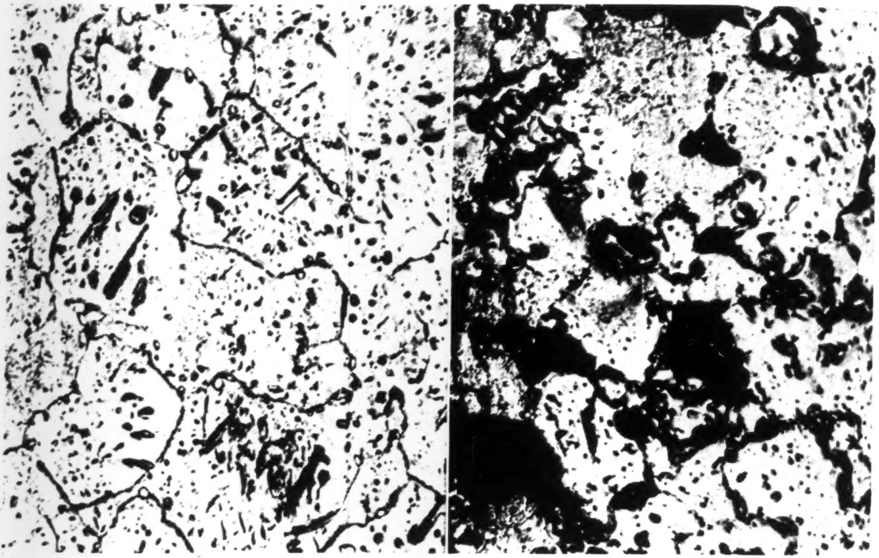
Etchant: Glacial acetic acid—30 per cent hydrogen peroxide.



(*a*) After failure in 2460 hr. at 730 psi. (*b*) After failure in 18,410 hr. at 370 psi.

FIG. 21.—Chemical Lead-0.9 per cent Antimony Sleeve (× 250).

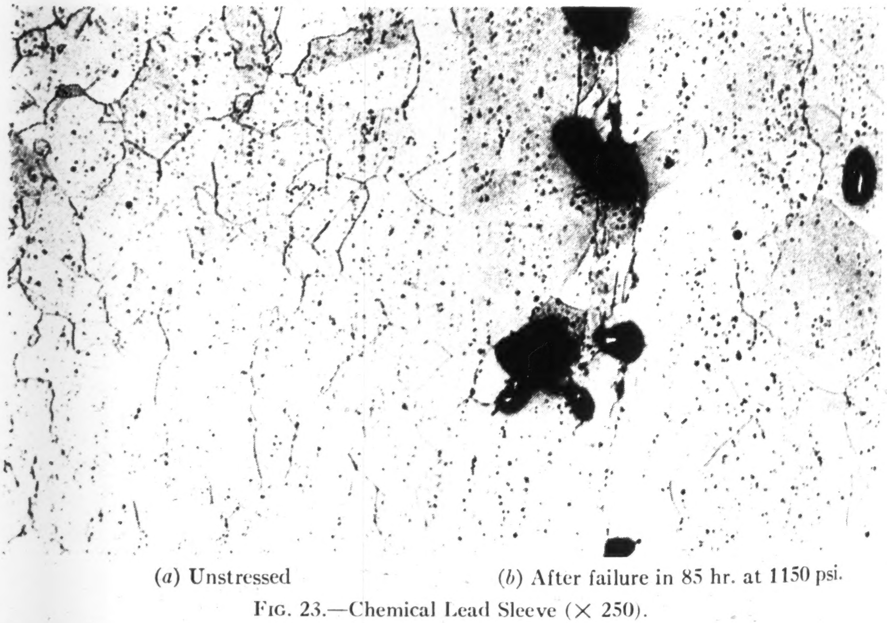
Etchant: Glacial acetic acid—30 per cent hydrogen peroxide.



(*a*) Unstressed (b) After failure in 98 hr. at 1630 psi.

FIG. 22.—Secondary Lead-3 per cent Tin Sleeve (× 250).

Etchant: Glacial acetic acid—30 per cent hydrogen peroxide.



(*a*) Unstressed (*b*) After failure in 85 hr. at 1150 psi.

FIG. 23.—Chemical Lead Sleeve (× 250).

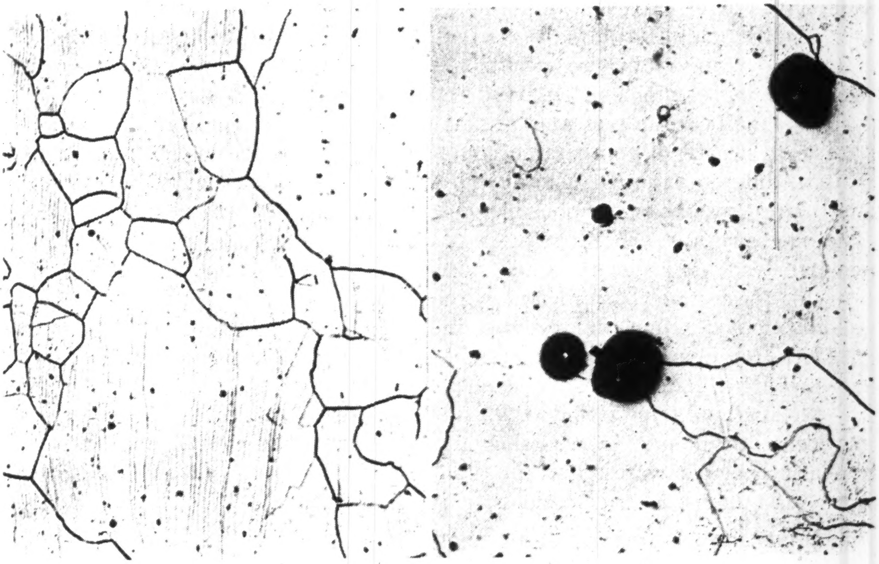
Etchant: Glacial acetic acid—30 per cent hydrogen peroxide.

partial recrystallization (presence of light grains and sharp boundaries) with the preponderance of grains still striated. Well-distributed antimony particles, typical of the extruded structure, are also evident. After 18,410 hr. the alloy is almost completely recrystallized and extensive agglomeration of the antimony is evident. While time is a factor in this phenomenon, the stress intensity, as well, is undoubtedly involved.

The typical structure of the unstressed lead-tin alloy (Fig. 22 (*a*)) consists of dark needles or spots which are composed of a tin-rich solid solution precipitated in the lighter lead-rich matrix. The nature of the creep failure is shown in Fig. 22 (*b*) for this alloy. The needles tend to coalesce and general recrystallization has taken place.

The microstructure of Chemical lead (Fig. 23) is quite different from the alloy structures. The dark spots in Fig. 23 (*a*) are primary copper particles aligned by the extrusion process. In Fig. 23 (*b*), the specimen which failed after the relatively short period of 85 hr. shows substantial grain growth.

Very little second phase can be seen in the structure of the detinned lead, Fig. 24 (*b*), but strain lines resulting from extrusion are evident. For the ruptured specimen, Fig. 24 (*b*), grain growth and local voids may be noted, not unlike those obtained in Chemical lead as shown in Fig. 23 (*b*).



(*a*) Unstressed (*b*) After failure in 1830 hr. at 530 psi.

FIG. 24.—Detinned Lead Sleeve (× 250).

Etchant: Glacial acetic acid—30 per cent hydrogen peroxide.

Tape Alloys:

The tensile data for the tape alloys listed in Table II show that Chemical lead is slightly stronger than high-purity lead. The improvement in tensile strength resulting from the addition of various percentages of tellurium, antimony, and calcium to these two basic leads also is indicated by the data in this table. Unlike the sleeving alloys, the tape alloys all showed a marked increase in tensile strength during aging at room temperature (80 to 85 F.). This increase ranged from approximately 10 per cent for Chemical lead to 30 per cent for some of the calcium alloys. These data do not reflect the maximum age-hardening characteristics of the lead-calcium alloys because the initial tests were made several years after extrusion, and these alloys age-harden most rapidly during the first few months following extrusion. Since the aging phenomena observed in the case of the tape samples were absent in the lead sleeves of comparable compositions, differences in extrusion conditions were probably responsible.

The stress-rupture characteristics of Chemical lead, high-purity lead, and alloys of these two basic leads with small additions of tellurium are shown in Fig. 15. This figure also contrasts the stress-rupture characteristics of Chemical lead with alloys containing 0.29 and 1 per cent antimony. Curves showing the relation between stress and the time required to produce 3 per cent elongation are shown in Fig. 16 for specimens of the same six tape alloys.

The stress-rupture characteristics of high-purity lead and alloys containing 0.02 and 0.027 per cent calcium are shown in Fig. 17, together with the stress-rupture characteristics of Chemical lead and four alloys of increasing calcium content. Curves showing the relation between stress and the time required to produce 3 per cent elongation are shown in Fig. 18 for these alloys.

A comparison of the creep properties of the tape alloys can be obtained by examination of Figs. 15 to 18 inclusive. These figures illustrate representative data selected from Tables IV and VI, and the groupings are such that a comparison with Chemical lead is readily made.

Chemical lead (Figs. 15 and 16) is superior to high-purity lead at all stresses. This superiority reflects the presence of small amounts of copper and silver in the Chemical lead. The addition of tellurium to either grade of lead resulted in an increased creep resistance at high stresses and a decrease at low stresses. It should be noted that this same behavior was found for antimony additions to Chemical lead in the case of the sleeving alloys. The effects of additions of antimony to Chemical lead (Figs. 15 and 16) were similar for the tape and sleeving alloys.

The creep characteristics obtained by alloying calcium with high-purity and Chemical lead are depicted in Figs. 17 and 18. Calcium in excess of 0.013 per cent, unlike the other alloying constituents, improves the creep resistance at all stresses. Unfortunately, in the installation of cable sleeves, it is necessary to "beat in" the ends. The increased stiffness of the lead-calcium alloys makes this operation difficult and this precludes their use as sleeving alloys at the present time.

All of the tape alloys showed measurable creep at a stress of 250 psi., not only for periods of 70,000 but also after 60,000 hr. It was impossible to evaluate the creep resistance of the specimens at 150 and 100 psi. without extrapolation. Attempts to extrapolate the data to these lower stresses led to inconsistent results, and this procedure is not recommended in the interpretation of creep data.

Exploratory tests made at 20 F. on Chemical lead, lead containing 0.033 per cent calcium, and lead containing 1 per cent antimony, indicate that such alloys have improved creep resistance at low temperatures (Figs. 9 and 11). The stress-rupture characteristics were improved without any measurable loss of ductility except for the 1 per cent antimony alloy.

CONCLUSIONS

1. There was no significant difference in the creep resistance of the sleeving alloys in a transverse or longitudinal direction.

2. Of the various sleeving alloys tested, the 0.9 per cent antimony and the 3 per cent tin alloys had the best resistance to creep at stresses in excess of 750 psi.; Chemical lead had the best creep resistance at lower stresses.

3. The secondary lead and detinned lead sleeves were found to be definitely inferior in creep resistance to Chemical lead sleeves. Likewise, the studies on the tape samples showed that high-purity lead was not as resistant to creep as Chemical lead.

4. Of the various sleeving alloys, Chemical lead sleeves were the only ones showing no measurable creep after 60,000 hr. and the maximum stress at which this condition prevailed lay between 100 and 150 psi.

5. Of the tape alloys, lead-calcium alloys with more than 0.013 per cent calcium had better creep resistance at all stresses than did the unalloyed Chemical lead.

6. The tellurium and antimony tape alloys were not as resistant to creep at low stresses as Chemical lead.

7. At low temperatures the creep resistance of lead alloys was improved materially.

8. The stress-rupture curves do not show a linear relationship between stress and the logarithm of time. Hence, no equation such as $S = S_1 (1 - c \log t)$ can be used with any assurance to extrapolate short-time creep data.

Acknowledgments:

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(10) J. N. Greenwood and H. K. Worner, "The Influence of Impurities on the Properties of Lead—The Influence of Tellurium on the Creep Rate of Commercial Lead," *Proceedings*, Australasian Inst. of Mining and Metallurgy, No. 101, pp. 57–87 (1936.)

(11) J. N. Greenwood and H. K. Worner, "Influence of Elements on the Properties of Lead—The Influence of Composition on the Creep Rate of Industrial Lead," *Proceedings*, Australasian Inst. of Mining and Metallurgy, No. 104, p. 385, December 31, 1936.

(12) A. A. Smith, "Creep and Recrystallization of Lead," *Transactions*, Am. Inst. Mining and Metalurgical Engrs., Vol. 143 (Inst. of Metals Division), p. 165 (1941).

(13) A. J. Phillips, "Some Creep Tests on Lead and Lead Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 170 (1936).

DISCUSSION

Messrs. H. F. Moore[1] **and C. W. Dollins**[1] *(presented in written form)*.—The authors of this paper have presented what is perhaps the most extensive series yet made of test results on these metals, especially for periods of loading above 1000 hr. The value of this paper to engineers and metallurgists interested in cable sheathing is very great.

Two points in the paper seem worthy of comment. In their Tables V and VI the authors report no appreciable creep after 60,000 hr. for certain alloys. In the creep tests made at the University of Illinois, creep was measured to 0.0001 in. over a gage length of 10 in., a considerably greater sensitivity of measurement than that used by the authors of this paper. It was found at Illinois that under loads as low as 100 psi. for the higher strength lead alloys and under loads of 50 psi. for the weaker lead alloys, definite indications of creep were obtained after 500 hr., or less under load.

The authors in their conclusion 8 and also in the body of the paper rather summarily dismiss the stress - log time formula given in *Bulletin 347* of the Illinois Engineering Experiment Station. It is quite true that this formula does not hold for very long times. This fact was pointed out in *Bulletin 347*, but the formula does give lower stress values than those found under tests lasting 1000 hr. or more. It gives results on the safe side, as is shown both by the tests of the authors and by tests at Illinois. It is believed that, while it is to be hoped that a more accurate formula will be developed from the results of these or of other tests, the stress - log time formula can be used with a good assurance that the results are on the safe side.

Mr. P. G. McVetty[2] *(presented in written form)*.—The authors have contributed to our knowledge of creep phenomena in general by making available the data contained in this paper. Only those who have made similar investigations can appreciate the large amount of work involved in collecting these data and in the subsequent interpretation and correlation.

It has been found useful to assume that the characteristics of lead and lead alloys at atmospheric temperature are similar to those of heat-resisting alloys at high temperatures. This assumption has permitted studies of flow characteristics in tests of shorter duration and without the complications of high temperature. It is somewhat disturbing to find the authors opposed to extrapolation from tests at higher stresses to the lower stresses used in design. This procedure is necessary in most cases involving heat-resisting alloys, unless we accept the alternative of more tests at low stresses and extremely long duration.

Mr. C. J. Snyder[3] *(presented in written form)*.—The authors' presentation of creep data on lead at low stresses is a much needed contribution to our literature. As a rule one has difficulty interpreting such data because different samples of apparently similar material sometimes give widely differing results, but the authors' data and interpretation give a very clear picture of the subject. However, I have a few questions and comments which I believe will be in order.

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[1] Member of the Staff of Talbot Laboratory, University of Illinois, Urbana, Ill.

[2] Manager, Strength of Materials Section, Research Labs, Westinghouse Electric Corp., East Pittsburgh, Pa.

[3] Engineer in Charge, Metallurgical Lab., Anaconda Wire and Cable Co., Hastings-on-Hudson, N. Y.

Can the authors explain why the tensile strength of the antimony alloy sleeves did not change more than shown in Table II? Antimonial lead sheaths on power cables usually show considerable change. For instance, the average strength of 28 samples of 0.8 per cent antimony alloy cable sheath, chemical lead base, was 3158 psi., when tested within 7 days after production; but 8 months later a spot test of six of the cables showed a strength of 4113 psi. Also the elongation of these samples had changed from an original of 38 per cent, as determined on a ring-shaped specimen, to 22 per cent. Other samples extruded as empty pipe from a cable covering press and water-cooled showed about the same change in 5 months. However, 5 yr. later the strength had dropped to 3440 psi. and the elongation had increased to 26 per cent. Other samples extruded from a pipe press and air cooled gave tensile values of 3650 psi. and 38 per cent elongation when 2 weeks old, but when 4.67 yr. old their values had changed to 4020 psi. and 30 per cent.

In the discussion, the authors pointed out that grain growth had taken place in the specimens of unalloyed lead cut from sleeves or tubes. Did they observe similar growth in the tape samples of unalloyed lead? My reason for asking this is that I thought the cold working of the sleeve samples due to splitting and flattening the tubes may have created strains in the specimens which caused them to recrystallize. I have never noticed grain growth when a tube specimen was used for creep test.

Were the authors able to distinguish and classify the character of the failures of all the specimens as to whether they were ductile type or intercrystalline type, such as shown by Bassett and Snyder?[4] We attributed considerable practical importance to that point because it appeared to be a form of stress cracking—perhaps stress-corrosion cracking— and it seemed that every time a material with good creep resistance was tested it was very sensitive to stress cracking. Such materials when used as cable sheaths have failed readily in slow bending in service.

My fourth point is an interesting experience, which also illustrates the sensitivity of lead specimens to history and handling. Creep tests were made on many ring-shaped specimens at various increasing hoop stresses between 90 and 500 psi. at 65 C. Specimens cut from samples extruded as empty tubes gave comparatively rapid creep at stresses up to 125 to 150 psi. At higher stresses, such as 175 to 200 psi., the creep rate was decidedly less than at 125 to 150 psi. and did not again equal the 125 psi. rate until a stress of about 300 psi. was reached. In other words, a plot of creep rate against stress gave a reversed Z-shaped curve. Specimens cut from samples extruded onto a cable core did not show the rapid creep at stress of 90 to 150 psi. In this low range the cable specimens were ten times better than tube specimens. However, above 300 psi. check rates were obtained. Later it was found that tube specimens which were stretched about 1 per cent over an expanding arbor would give values similar to cable specimens at all stresses.

MR. H. E. HOWE[5] (*by letter*).—The data obtained in our laboratory on the creep of lead and lead alloys have also shown the superiority of the copper-bearing acid and chemical leads over common and high purity lead at all stresses, as well as over 1 per cent antimonial lead, tin-lead alloys, and tellurium-lead at the lower stresses. Creep results for three alloys—acid lead, chemical lead, and a 2 per cent tin alloy commercially extruded as cable sheathing—are plotted in the accompanying Fig. 25.

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[4] W. H. Bassett, Jr. and C. J. Snyder, "The Testing of Lead Cable Coverings," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 910 (1940).

[5] Research Dept., American Smelting and Refining Co., Barber, N. J.

The authors suggest that the superiority of the chemical lead reflects the presence of small amounts of copper and silver. In tests on copper-bearing leads containing less than 0.002 per cent silver or silver in higher amounts than are present in chemical lead, we have not detected any improvement in properties due to the presence of the silver. The effect of silver is definitely minor and appears to be obscured by the more predominant effect of the copper.

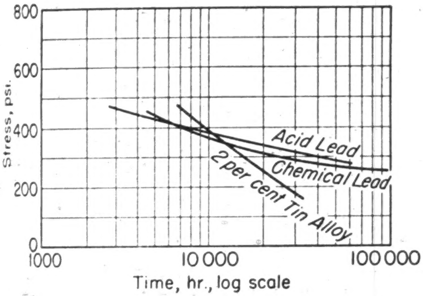

FIG. 25.—Creep Resistance of Cable Sheathing at 3 per cent Elongation.

We have conducted many tests on laboratory and commercially extruded alloys and have found that extrusion procedure has an appreciable effect on tensile strength, aging characteristics, and creep rates. Such variations further limit the reliability of extrapolating from higher to lower stresses.

MR. G. M. BOUTON (*author*).—The use of Mr. Moore's formula for extrapolating the stress-rupture data is not possible, since the formula is represented by a straight line when plotted on a semi-logarithmic basis and the data presented in this paper definitely fall on a curve. While it is true that the values obtained by the use of the formula are on the safe side, they do not permit appraisal of the various alloys. As an extreme example, extrapolation of the 100- and 1000-hr. values on several of the materials would indicate rupture at zero stress in finite times.

There are sound reasons why no formula could be expected to fit creep curves of lead alloys. In the first place, lead alloys are not constant in properties during aging. Precipitation hardening may take place and the rate at which precipitation takes place is a function of deformation of the material. The degree to which the effect of deformation affects precipitation may be different for different alloys and may even be dependent on the extent to which the dispersion process has taken place at the time an individual alloy is placed on test. In addition to the precipitation changes, different lead alloys work-harden at different rates and recrystallize at room temperatures after different amounts of deformation. Thus an alloy stressed at a high value might deform and work-harden. The creep rate would then depend on whether the alloy would recrystallize. At lower stresses, where the amount of deformation is less and the time factor is greater, the alloy might behave much differently. Also, at one stress, elongation might take place by grain deformation while at another stress, grain boundary separation might be more pronounced. Any equation which would predict behavior at low stresses from those at high stresses would have to integrate all of these factors. Because of this complexity it would seem, therefore, that the hope of obtaining such an equation for any but the simplest alloys is rather remote.

Mr. Moore's comments concerning the use of more sensitive methods for measuring creep are of considerable interest. Since, in most applications, only slight creep is permissible, it should be possible to determine a creep rate at low stresses in relatively short periods of time which might be extrapolated to predict the time required to produce 1 per cent or less extension. Since the deformation is small, it should have much less effect on precipitation, recrystallization and other factors that influence the general properties of the materials.

While there are many reasons why there is little hope for a formula to predict the behavior of lead at low stresses from tests made at high stresses, this situation may not be true of all materials. The heat-resisting alloys mentioned by Mr. McVetty may not undergo the changes observed in lead alloys, in which case extrapolation by formula might be possible.

We are pleased to receive the confirming evidence presented by Mr. Howe. We agree entirely on the large effects that production conditions may have on the properties of the various lead alloys. Mr. Snyder's comments on lead-antimony sheath serve further to emphasize the effect of production conditions on age-hardening alloys. The hardening and softening phenomena cited are characteristic of lead-antimony cooled at certain rates. Quenching accentuates the age-hardening effect and cooling over a long period minimizes it. As stated in the paper, the sleeves were allowed to cool in air.

The change in grain size of the pure lead tape samples was not determined. Since they were produced under conditions designed to simulate cable sheath manufacture, involving reeling, they were probably not stress free.

Indications of the ductility of the various materials may be obtained from the elongations at failure shown in Figs. 5 to 11, inclusive. Generally speaking, the samples having the lowest elongation had the most intercrystalline fractures.

The instances of anomalous creep behavior cited may be associated with work hardening at the time that loads are applied to the samples. The initial set of the samples stressed at intermediate values may have resulted in a low subsequent creep rate.

THE BRINELL HARDNESS OF GRAY CAST IRON AND ITS RELATION TO SOME OTHER PROPERTIES*

By J. T. MacKenzie[1]

Brinell Hardness and Tensile Strength

During the past few years there has been an increasing tendency to introduce a maximum value for Brinell hardness in specifications for gray cast iron which call for a minimum tensile strength. This is a simple, convenient, and reasonable method of obtaining the desired machinability without using the dubious alternative of putting a maximum on the strength, since it is hardly conceivable that a casting could be too strong if the unnecessary extra strength is obtained without sacrificing other desirable properties. Navy Department Specification 46-I-5C carries such a specification with the following limits:

Tensile Strength Minimum, psi.	Brinell Hardness Maximum
30 000	250
35 000	260
40 000	270
45 000	280

These values are indicated in Fig. 2. In order to have some reliable criteria for setting up such specifications, A.S.T.M. Committee A-3 on Cast Iron asked the author to make a study of the available data and present it in condensed form so that consumer and producer could see whether any given limitation of hardness was or was not within reason for the tensile strength required.

The following firms contributed test results:

American Cast Iron Pipe Co.
Buffalo Foundry and Machine Co.
Climax Molybdenum Co.
Cooper Bessemer Corp.
Deere and Co.
Farrell Birmingham Co.
International Harvester Co.

A great deal of the data is from experimental heats, but much more from actual operations. Most of the experimental heats were average results from duplicate (or more) bars and the operating results usually included from ten to fifty readings from as many bars of each grade of iron being made. A total of 1553 sets were submitted showing both tensile strength and Brinell hardness. These were on the following size bars:

Bar A (0.875 in.)	145
Bar B (1.20 in.)	982
Bar C (2.0 in.)	171
3 to 10 in.	46

Other results were from bars cut from actual castings.

The data for tensile strength and Brinell hardness for all tests are shown in Fig. 1. The area outlined by the heavy lines contains all but eight of the results and will in the subsequent discussion be called the field. The eight results omitted were the four at the right which had extremely high manganese or molybdenum and the two with Brinell hardnesses of 86 to 89 which were from a very heavy casting that had been in service over thirty years, presumably under considerable stress.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] Chief Metallurgist, American Cast Iron Pipe Co., Birmingham, Ala.

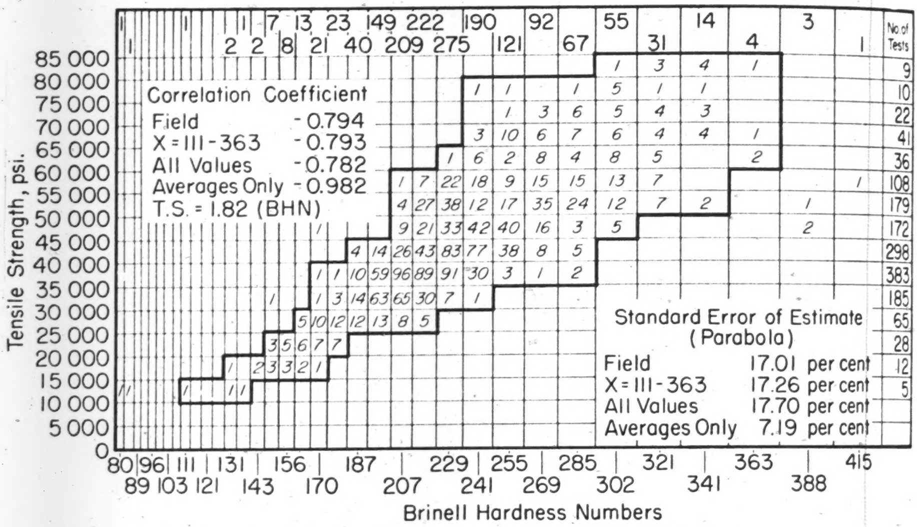

Fig. 1.—Data for Brinell Hardness-Tensile Strength Tests.
1553 tests representing all companies.

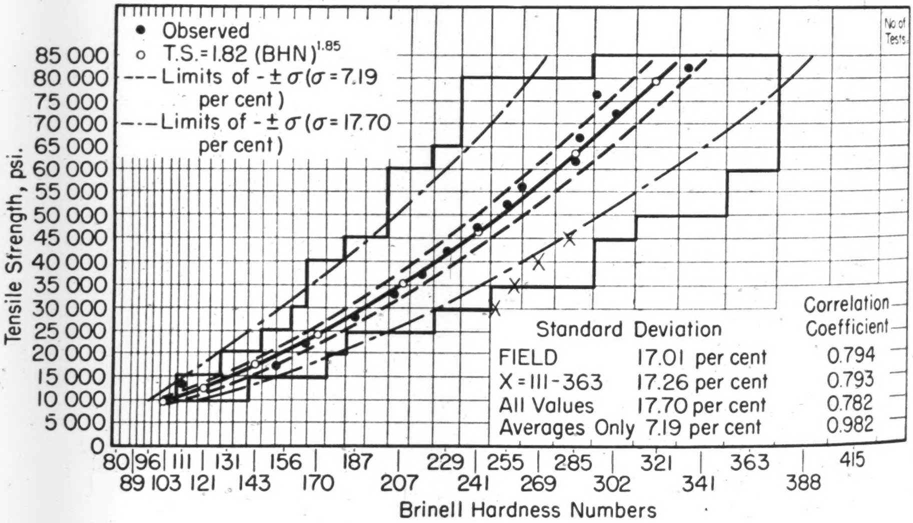

Fig. 2.—Statistical Analysis of Data in Fig. 1.
1553 tests representing all companies and all sizes of specimens.

The data in Fig. 1 are analyzed statistically in Fig. 2 which shows the field, the average values of both tensile strength and Brinell hardness for each tensile strength class (steps of 5000 psi.), the equation of best fit calculated from the 1553 individual values by least squares, and the standard error of estimate and the correlation coefficient using the average value for each cell in Fig. 1 but giving the cell its full weight according to the number of tests.[2] The standard error and correlation coefficient are shown for the field; for all Brinell values from 111 to 363; for all values; and, for the class averages only (the black dots). The Navy Specifications, 46-I-5C, are also indicated. Several other equations were tried: the best straight line equation (least squares)

$$\text{Tensile strength, psi. (in thousands)} = 0.38\ \text{BHN} - 44.6$$

fits quite well from class 25 and above, but the standard error is 23.5 per cent and calculated tensile strengths are much too low in the lower range.

The best hyperbola equation (least squares)

$$\text{Tensile strength, psi. (in thousands)} = \frac{46\ (\text{BHN})}{480 - (\text{BHN})}$$

also fits well from class 25 on up, but gives tensile values too high in the lower groups.

TABLE I.—EXPECTED TENSILE STRENGTH CALCULATED FROM BRINELL HARDNESS NUMBER.

Tensile Strength = 1.82 (BHN) $^{1.85}$ for Gray Cast Iron

Diameter, in.	BHN	Tensile Strength, psi.
3.20	363	99 100
3.30	341	88 200
3.40	321	78 900
3.50	302	70 500
3.60	285	63 300
3.70	269	56 900
3.80	255	51 500
3.90	241	46 400
4.00	229	42 200
4.10	217	38 200
4.20	207	35 000
4.30	197	32 000
4.40	187	29 000
4.50	179	26 800
4.60	170	24 300
4.70	163	22 500
4.80	156	20 800
4.90	149	19 200
5.00	143	17 500
5.10	137	16 300
5.20	131	15 000
5.30	126	14 000
5.40	121	13 000
5.50	116	12 000
5.60	111	11 100
5.70	107	10 300
5.80	103	9 200

TABLE II.—SUMMARY OF THE BRINELL HARDNESS-TENSILE STRENGTH DATA.

Tensile Class, psi. × 10^{-3}	Brinell Hardness Number	Average Tensile Strength	Average Brinell Hardness Number	Standard Deviation Brinell Hardness Number	Standard Deviation,[a] per cent of average Brinell Hardness Number
10 to 14.9	5	13.1	111	21	19
15 to 15.9	12	17.7	153	11	7
20 to 24.9	29	22.6	166	10	6
25 to 29.9	65	27.9	187	16	8
30 to 34.9	185	33.1	204	12	6
35 to 39.9	383	37.4	216	16	7
40 to 44.9	297	42.6	230	19	8
45 to 49.9	172	47.0	243	27	10
50 to 54.9	179	52.3	255	33	13
55 to 59.9	108	56.9	262	35	13
60 to 64.9	36	62.3	285	33	12
65 to 69.9	41	67.3	286	32	11
70 to 74.9	22	72.4	302	25	8
75 to 79.9	10	76.9	294	29	10
80 to 84.9	9	82.5	335	19	6

[a] These deviations are in per cent of the average value of Brinell Hardness for each class as shown above. The deviations shown on Figs. 1 and 2 are strictly termed the Standard Error of the Estimate, and are in terms of per cent deviation of tensile strength values about the curve of best fit.

Table I gives values for the tensile strength according to the parabola

$$\text{Tensile strength} = 1.82\ (\text{BHN})^{1.85}$$

calculated for each tenth of a millimeter

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[2] The standard error of the estimate is a measure of the extent of the error in estimating ordinate values from given abscissa values by means of a fitted curve. The deviations of individual y values from the expected values were calculated as *percentage* deviations in order to permit comparison in the same units of the standard errors of several estimated curves and sets of data.

$$\sigma_{\text{est.}}\ (\%) = \sqrt{\frac{\sum\left[\frac{100\ (y - \bar{y})}{\bar{y}}\right]^2}{N}}$$

where:

σ_{est} = standard error of estimate,
y = individual ordinate values,
$\bar{y}$ = predicted y value for a given x value, and
N = number of values.

Thus, σ_{est} serves as a measure of the dispersion of values about a central curve and consequently as a means of comparing the goodness of fit of different curves for a given set of data.

The correlation coefficient is a measure of the proportion of the variation in one variable which is associated with another variable.

$$r = \frac{\Sigma xy - N\bar{x}\bar{y}}{\sqrt{[\Sigma(x^2) - N\bar{x}^2]\ [\Sigma(y^2) - N\bar{y}^2]}}$$

where:

r = correlation coefficient,
x and y = individual values,
$\bar{x}$ and $\bar{y}$ = mean values, and
N = number of pairs.

The correlation coefficient, r, is a pure number which may vary between +1 and −1. Units indicate perfect correlation, zero no correlation, and the sign shows the direction of the slope of the best straight line.

of diameter of impression from 3.2 to 5.8.

average of the Brinell and the tensile strength.

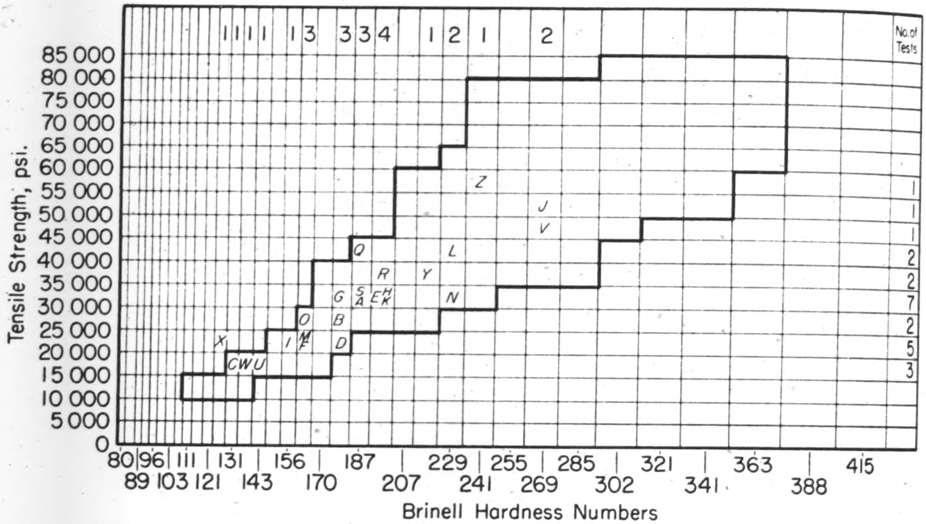

Fig. 3.—Data from the 1933 Report of Subcommittee XV on Impact Testing of A.S.T.M. Committee A-3 on Cast Iron.
24 tests.

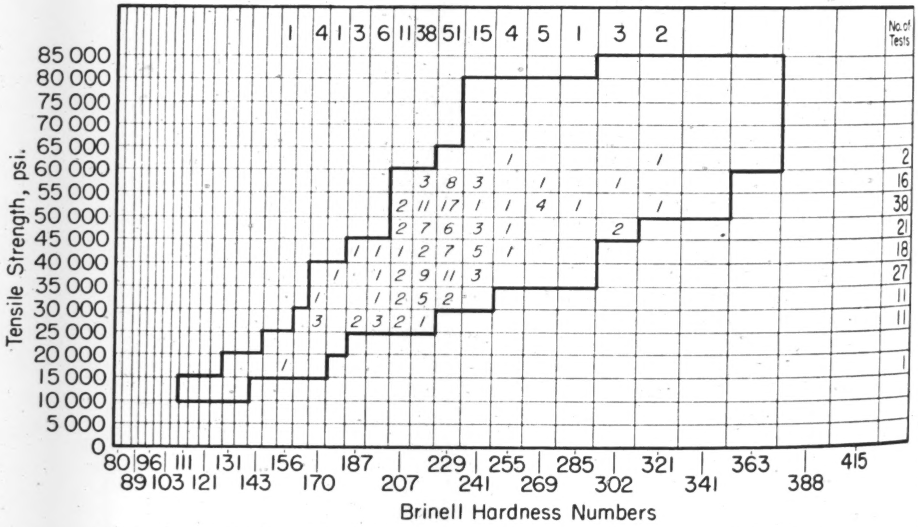

Fig. 4.—Data for Brinell Hardness-Tensile Strength Tests on $\frac{7}{8}$-in. Bars.
145 tests.

Table II shows the standard deviation of the Brinell hardness numbers, both in numbers and in per cent of the average for each class, together with the actual

To illustrate the usefulness of the field, the data from the 1933 Report[3] of Subcommittee XV on Impact Testing of A.S.T.M. Committee A-3 on Cast Iron are plotted in Fig. 3. All of the twenty four irons, except the austenite iron X, are in the field—even the martensitic iron N which, together with the 2 per cent phosphorus iron, is on the bottom line. Since these are by far the most carefully obtained values available, the confirmation of our present data is very satisfying.

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[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part I, p. 87 (1933).

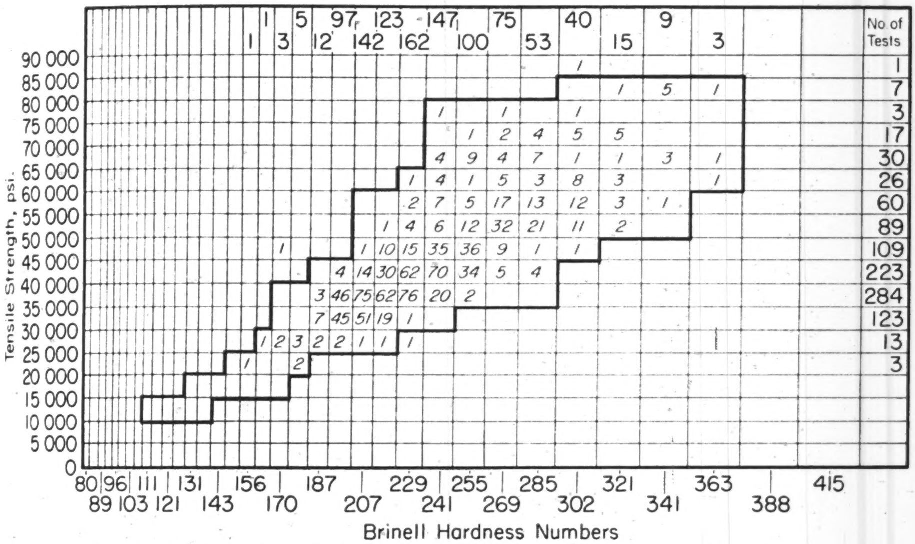

FIG. 5.—Data for Brinell Hardness-Tensile Strength Tests on 1.2-in. Bars. 988 tests.

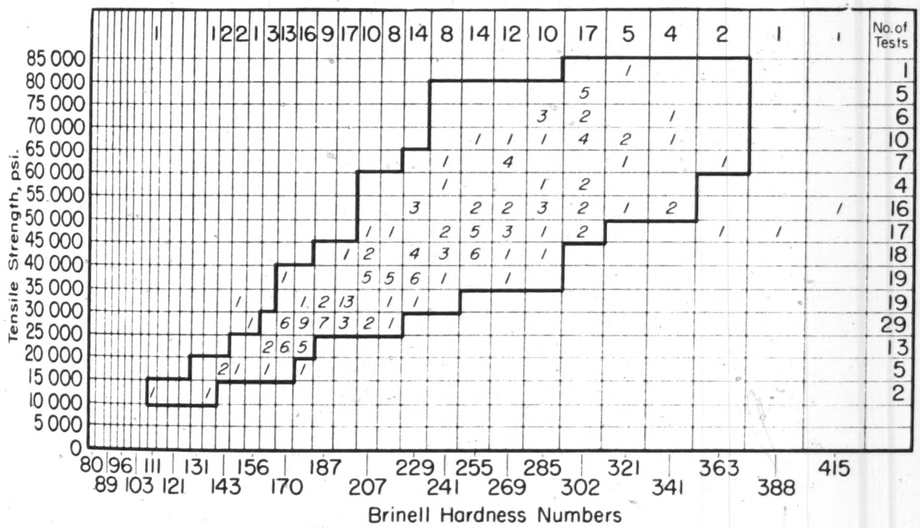

FIG. 6.—Data for Brinell Hardness-Tensile Strength Tests on 2-in. Bars. 171 tests.

There is no *a priori* reason to assume

that the Brinell hardness number-tensile strength relation would change with section size, but the sizes were plotted separately to be sure. The results are shown in Figs. 4, 5, 6, and 7. There is no difference to the eye (no calculations were made) except in the case of the 2-in. bars, but this is not due to a section effect but to a large number of bars verging on (if not actually in) the martensitic condition and usually heavy in manganese or molybdenum.

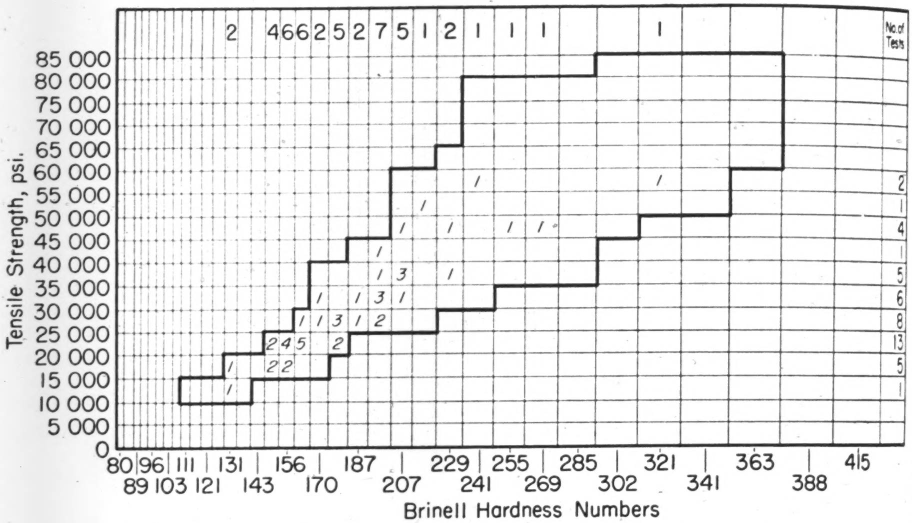

Fig. 7.—Data for Brinell Hardness-Tensile Strength Tests on 3-in. and Larger Bars.

46 tests.

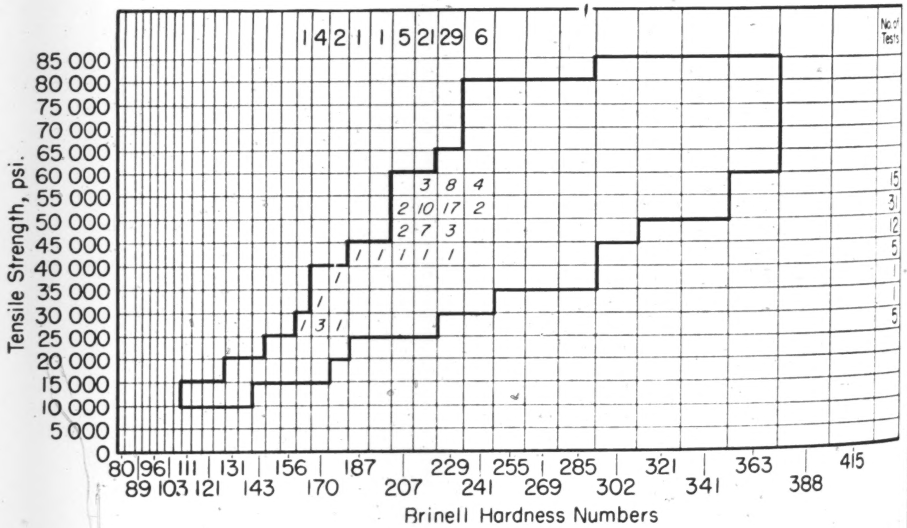

Fig. 8.—Tests on Inoculated Irons from Cooper-Bessemer Corp.

70 tests.

A study was made of the elements Ni, Cr, Cu, Mo, Mn, and P. Some hundred heats of white irons were examined and all fell in the lower right of the diagram completely out of the field. Irons with over 2 per cent of copper, 3 per cent nickel, and 1 per cent phosphorus were invariably on the low side, unless well balanced or heavily inoculated. A study was made of inoculated and untreated irons. A strong tendency from inoculation toward the upper side of the diagram was noticeable—the results from one foundry were especially good and are shown in Fig. 8, though some other inoculated irons were fairly close to the average for all groups. These were also low-phosphorus and low-alloy irons. Barlow and Lorig's[4] results when plotted on the field show on the high side for the graphitizing inoculant, down the middle for the stabilizing type, and decidedly on the low side for the untreated irons. (Their results are not a part of the data in Fig. 1.)

Brinell Hardness Number and Bending Strength

The author was also requested to examine the data for other correlation with Brinell hardness number, especially the transverse test. In the bars submitted, there were 1023 bars with transverse as well as tensile strength and Brinell hardness data.

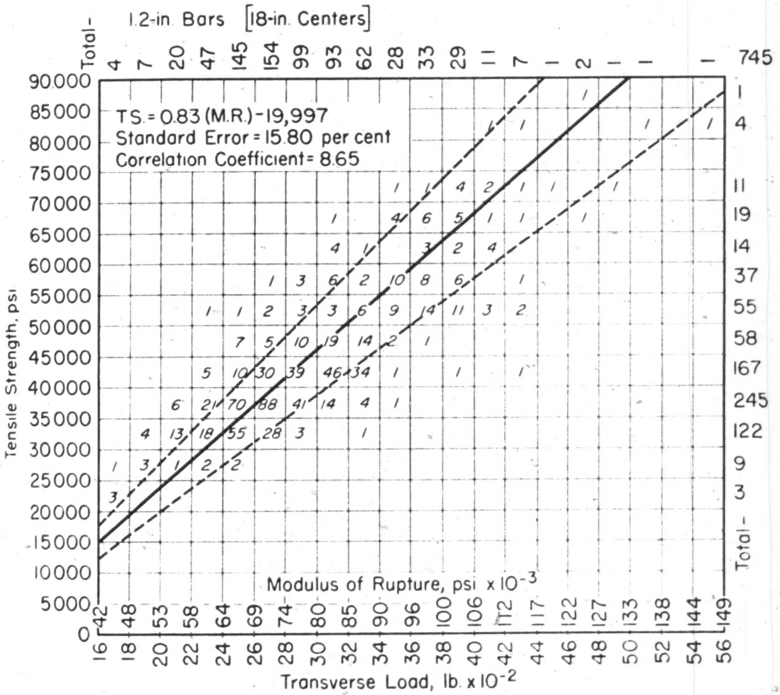

Fig. 9.—Tensile Strength *versus* Modulus of Rupture Data for the 1.20-in. Bar in Detail with the Best Straight Line by Least Squares and the Standard Error Drawn in.

These were in sizes as follows:

Bar Dimensions			Number of Tests
Diameter, in.	Span, in.	Span-Depth Ratio	
0.875	12	14	81
1.20	18	15	745
2.0	24	12	158
3.0	24	8	39

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[4] Tom Barlow and C. H. Lorig, "Gray Cast Iron—Tensile Strength, Brinell Hardness and Composition Relationships," *Am. Foundryman*, September, 1945.

Since the tensile strength-Brinell hardness relation was already satisfactorily established, it was only necessary to determine the ratio of transverse strength to either one, and the most convenient seemed to be the tensile strength, since the units could be expressed as unit stress in each case. Figure 9 shows the data for the 1.20-in. bar B in detail with the best straight line by least squares and the standard error drawn in. Figure 10 shows the best straight lines for all sizes, from which it is obvious that the tensile strength increases faster per unit of transverse stress, the smaller the bar. The sudden change in slope from the 2 to the 3-in. bars may be due to the smaller span-depth ratio of the 3-in. bar.

The data on the different sizes may be summed up in the statistics shown in Table III, which gives the best straight line, the standard error, and the correlation coefficient for each size using the individual results, and for the average of all sizes using only the average transverse results for each tensile class.

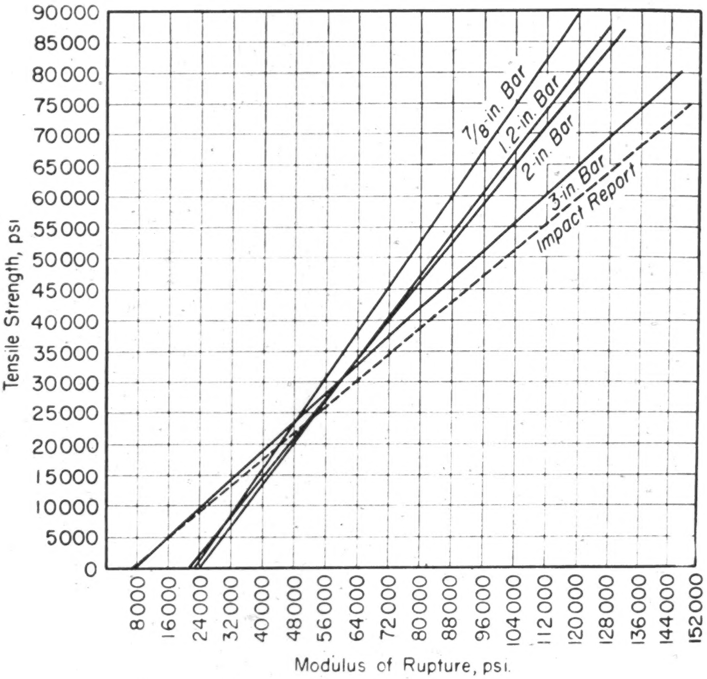

FIG. 10.—Tensile Strength *versus* Modulus of Rupture for All Sizes of Specimens.

TABLE III.—RELATION OF TENSILE TO TRANSVERSE STRENGTH.

Equation, Tensile Strength = $m \times$ Modulus of Rupture − b

Size, in.	Equation Constants		Standard Error, per cent	Correlation Coefficient, r
	m	b		
0.875	0.92	21 200	16.65	0.804
1.20	0.83	20 000	15.80	0.865
2.0	0.79	17 100	15.42	0.935
3.0	0.57	4 100	13.24	0.946
All sizes, (averages)	0.79	16 600	10.69	0.990
Impact Report[2]	0.56	5 000	9.89	0.975

The data from the impact report are also included in the table. It should be remembered that the bars in the impact investigation were machined down from 1.4 to 1.20-in. diameter.

Brinell Hardness and Stiffness

The next relationship of interest was that of hardness and stiffness. Since few data on deflection were available, except at the breaking point, it seemed that the tests from the impact report alone would furnish a reasonably good approximation.

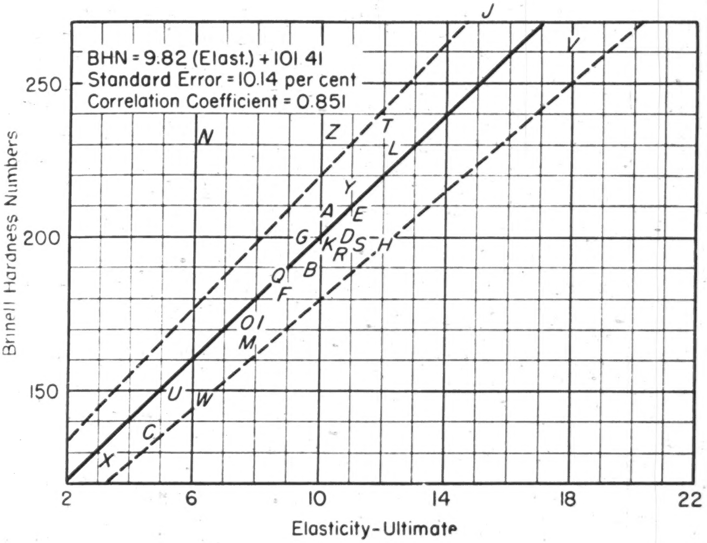

Fig. 11.—Data for Brinell Hardness-Stiffness Tests, Ultimate Elasticity.

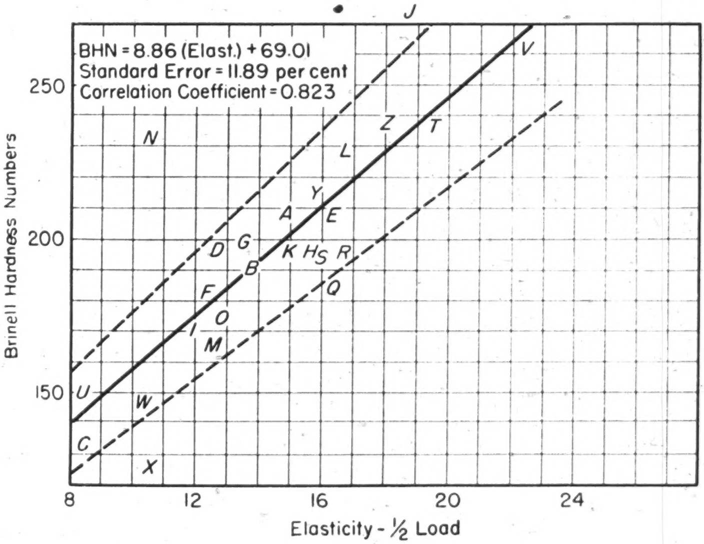

Fig. 12.—Data for Brinell Hardness-Stiffness Tests, One-Half Load Elasticity.

Figures 11 and 12 show these data (which include the two specimens that are not really gray irons, X and N). The correlation is very good and the error small. If X and N were omitted, the agreement would be much better; but since as-cast bars could not be expected to be nearly so consistent as these machined bars, they were retained for the present purpose. Elasticity was used in millions of pounds per square inch to simplify calculations.

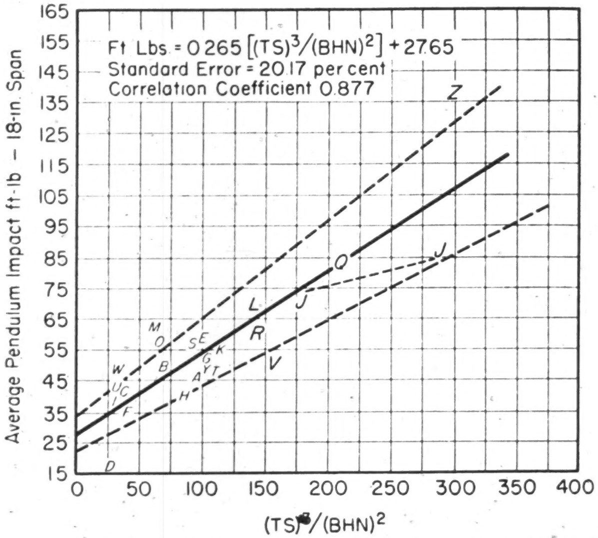

FIG. 13.—Data for Brinell Hardness-Impact Tests, from A.S.T.M. 1933 Impact Report, Using Drop Test.

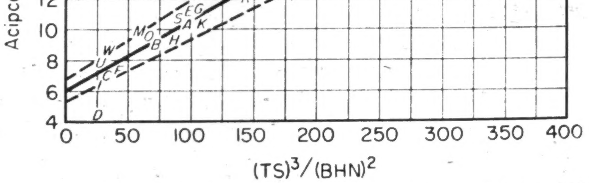

FIG. 14.—Data for Brinell Hardness-Impact Tests, from A.S.T.M. 1933 Impact Report, Using Pendulum Method.

BRINELL HARDNESS AND IMPACT STRENGTH

Since it has been shown that the resistance to shock is a function of the resilience, that is, the area below the stress-strain curve, it seemed probable that the tensile-Brinell hardness ratio would also be an index of the toughness. The simple ratio tensile strength-Brinell hardness was tried on the impact data with fairly good results, but it seemed more likely that some higher power of the tensile strength would be more logical. The ordinary formula for transverse resilience (MR^2/E) suggested the square of the tensile strength divided by the Brinell hardness number, but this seemed to overemphasize strength as compared to stiffness and neglected the increased curvature of the stress-strain curve in the softer irons. An intermediate stage of the cube of the tensile strength divided by the square of the Brinell hardness number was therefore investigated and proved slightly better in correlation as shown in Table IV, but all of them showed fairly good correlation with both the drop test and the pendulum, though as would be expected, the drop test correlates better since it is nearer to the triangular resilience than the pendulum test. The results are plotted in Figs. 13 and 14.

TABLE IV.—RELATION OF IMPACT PROPERTIES TO VARIOUS TENSILE STRENGTH-BRINELL HARDNESS NUMBER RATIOS.

Data from the 1933 Impact Report.

Drop Test—1.20 diameter, 6-in. span, ½-in. increment, 25-lb. hammer.

Pendulum—Charpy type, 1.20 diameter, 18-in. span.

Drop Test vs	Equation Constants[a]		Standard Error, per cent	Correlation Coefficient
	m	*b*		
Tens/BHN	0.105	6.7	12.4	0.929
Tens^2/BHN	0.112	4.4	11.9	0.943
$\text{Tens}^3/\text{BHN}^2$	0.046	6.0	12.2	0.949
Pendulum Impact vs				
Tens/BHN	0.305	4.6	24.17	0.847
Tens^2/BHN	0.629	19.7	19.15	0.827
$\text{Tens}^3/\text{BHN}^2$	0.265	27.7	20.17	0.877

[a] Equation:

Drop (or Pendulum) Impact Strength = $m\ (\text{tens}^x\ \text{BHN}^y) + b$

TABLE V.—DROP TEST VERSUS TENSILE STRENGTH-BRINELL HARDNESS NUMBER RATIOS.

Number of sets—B bars 59, C bars 54.

Each set is the average of three tension tests and from six to ten drop tests.

Equation—Drop Test = $m\ (\text{tens}^x\ \text{BHN}^y) + b$.

Ratio	Equation Constants		Standard Error, per cent	Correlation Coefficient
	m	*b*		
Bar B: 1.20-in. diameter, 6-in. span, 1-in. increment, 25-lb. hammer				
T.S./BHN	0.049	0.294	19.87	0.535
$\text{T.S.}^2/\text{BHN}$	0.056	4.43	19.13	0.602
$\text{T.S.}^3/\text{BHN}^2$	0.019	5.99	22.29	0.401
Bar C: 2.0-in. diameter, 12-in. span, 1-in. increment, 50-lb. hammer				
T.S./BHN	0.0897	0.015	15.55	0.643
$\text{T.S.}^2/\text{BHN}$	0.1014	8.81	14.70	0.674
$\text{T.S.}^3/\text{BHN}^2$	0.045	10.38	16.27	0.602

Drop test results from one foundry were available for 59 B bars and 54 C bars which were analyzed with the results shown in Table V. These data compare very poorly with the results from the impact report,[3] but the bars were not machined; thus, the condition of the surface is a highly important factor in the drop test but does not affect the tensile strength or Brinell hardness at all. In both sets, the square of the tensile strength divided by the Brinell hardness number is a better correlation than the other ratios.

The height of drop varied from 5 to 15 in. on the B bars, and from 8 to 20 in. on the C bars. (The abscissa units are actually correct only for the T.S./BHN ratio. $\text{T.S.}^2/\text{BHN}$ units are multiplied by 10^{-5} and the $\text{T.S.}^3/\text{BHN}^2$ units are multiplied by 10^{-7}.)

The charts for $\text{T.S.}^3/\text{BHN}^2$ are shown in Figs. 15 and 16. Assuming that this is the best ratio when the same metal is being tested (as in the 1933 Report[3]) then the correlation coefficients show that the drop test is affected by other factors than the tensile strength and Brinell hardness of the metal some ¼ to ½ in. under the skin. The extent of this is statistically the difference between unity and the square of the correlation coefficient: in the case of the 1.20 bars:

$$1 - (0.4)^2 = 0.84$$

and for the 2-in. bars

$$1 - (0.6)^2 = 0.64$$

That is, 84 per cent and 64 per cent, respectively, of the variation in impact strength is due to factors other than tensile strength and Brinell hardness. In spite of the rather unsatisfactory results of this study on impact, it is quite clear that with a given tensile strength we may expect more toughness from the lower hardness numbers, or to revert to Fig. 1, the tough irons are on the left of the field. No Ni-resist irons were included except in Fig. 3 (iron X), but some half-dozen sets ranging from 18,000 to 30,000 psi. tensile strength were checked and are all outside of and to the left of the field and they are all very tough.

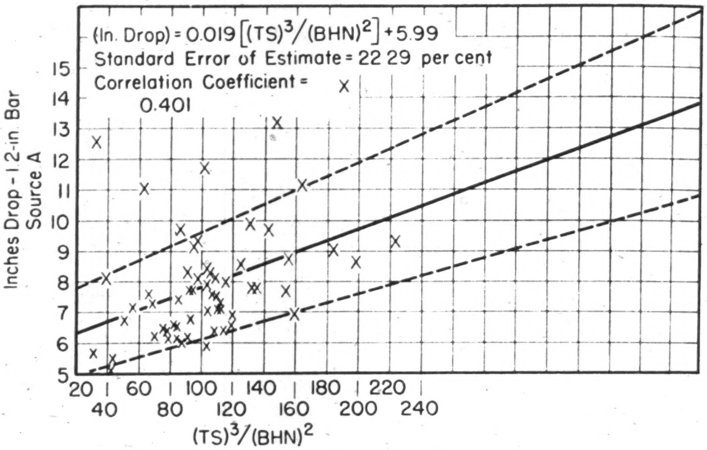

Fig. 15.—Chart for Drop Tests Using 1.2-in. Bar A, for Ratio $(T.S.)^3/(BHN)^2$.

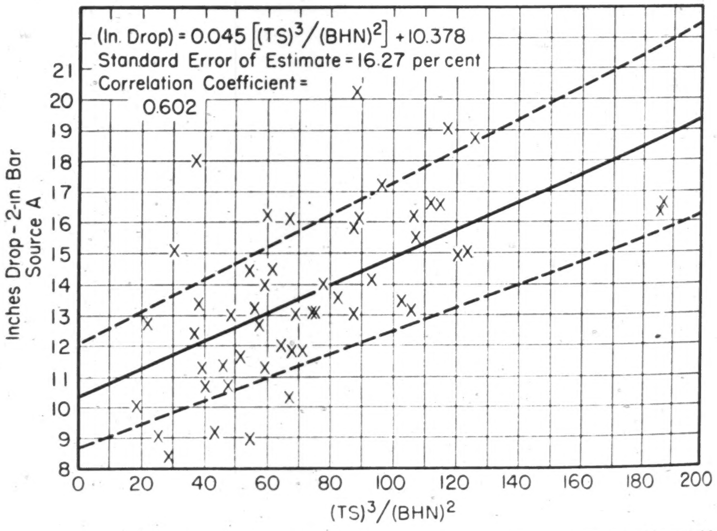

Fig. 16.—Chart for Drop Tests Using 2-in. Bar A, for Ratio $(T.S.)^3/(BHN)^2$.

One reason for the scatter of the tensile strength-Brinell hardness data is that there was no uniformity in the place the readings were taken. Some laboratory workers ground a flat on the side of the transverse bar; some read half way between edge and center on the ground end at the fracture; one took readings at center half way, and edge and averaged these; while several used the tension specimen after fracture, grinding off the threads for a flat sufficient for the impression. Since these latter were in the majority and it is perhaps the simplest and easiest method, the author would suggest that this method be standardized by Committee A-3.

DISCUSSION

Mr. S. C. Massari.[1]—The author is to be complimented on the very excellent mode of presentation of the large amount of data covered by this paper, which, as a consequence, offers little opportunity for discussion.

In the opening paragraph, the statement is made that minimum tensile strength and maximum value for Brinell hardness in specification is a simple, convenient, and reasonable method of obtaining desirable machinability without using the dubious alternative of putting a maximum on the strength. It would appear that there are cases in which maximum strength may also be of importance, as for example, in designing a group of components, should one of the components be intended to fail before the others, either for economic reasons or ease of replacement. Similarly, damping capacity is closely related to graphite size, and distribution may not be protected by such a specification. Superficial hardness may not in itself be a reliable index of machinability.

It is believed that the excellent statistical analysis of the data involved would have been further enhanced had at least typical methods of computation been included to aid those not so thoroughly versed in these methods.

It is unfortunate that full agreement on the location for Brinell hardness measurements had not been established at the beginning of this investigation, as it is quite likely that the relations would have been in even closer agreement. Similarly, in the case of the very large tension test bars, no mention is made of the size of the machined bar nor mention of the exact location in the section of the casting from which certain bars were cut. The inclusion of this information would prove interesting.

Mr. R. G. McElwee.[2]—I should like to add my own comment that I think what Mr. Massari had in mind in selecting an iron of considerable section sensitivity was that there might have been a considerable spread of Brinell hardness in the bar itself. In other words, if a specimen were taken from the side rather than the center of a large bar, it may have had a Brinell hardness of one value on one side and another on the other which may be a little misleading.

While I was unable to supply any bars to Mr. MacKenzie for this investigation, I did discuss it with him. It was our experience also that inoculated bars did have a relatively high strength-hardness ratio. In fact, we discussed some in which that ratio was in the neighborhood of 250 which was our measure of the virtue of the iron. That is also true of alloyed bars in which inoculation followed the alloy.

Mr. J. S. Vanick.[3]—I looked over some of the diagrams that Mr. MacKenzie has drawn up, particularly from the standpoint of the original problem of studying the spread in hardness for any specific tensile strength range. While the author's statistical analysis covers the ground very thoroughly, I thought I should find something of a practical nature for outlining a hardness specification. Figure 4 shows that for irons possessing 30,000 to 60,000 psi. tensile strength, practically all of them fall within a spread of one hundred units Brinell on the block diagrams. In the case of the $\frac{7}{8}$-in. diameter bar, 90 per cent of the tests would fall within a 40 point Brinell hardness spread for any specific tensile strength in the Nos. 30, 40, and 50 classes of iron (A.S.T.M. Specifications A–48).[4]

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[1] Technical Director, Am. Foundrymen's Assn. Chicago, Ill.

[2] Manager, Foundry Alloy Div., Vanadium Corporation of America, Detroit, Mich.

[3] Metallurgist, The International Nickel Co., Inc., New York, N. Y.

On the 1.2-in. bar of Fig. 5, something like 80 per cent of the hardness values fall within a 50 point hardness spread. Of the remaining 20 per cent, 5 percent are on the soft side and 15 per cent are on the hard side. These results mean to me that foundrymen are quite conscious of the need to reduce what might become a scrap loss due to hardness values that are out of bounds. It should not be too difficult to improve foundry control to the extent of getting the hardness for a 35,000 psi. tensile strength iron inside the brackets of a 50 point Brinell hardness spread, as, for example, a minimum of 180 and a maximum of 230.

The other point that occurred to me from what Mr. MacKenzie has also emphasized, is that as the sections grow larger, the hardness values for any tensile strength spread wider. That means more work for us in the foundry in studying the causes for such dispersions.

MR. J. E. HURST[5] (*by letter*).—The paper by Mr. MacKenzie, as far as my knowledge goes, is the first study of this sort to be published in connection with cast iron. The statistical treatment indicates a definite correlation between the Brinell hardness and the tensile strength in cast iron. It is not clear to me that this relationship will provide a method in which the Brinell hardness can be used as an indication of the strength of cast iron as is often done in the case of steel. The difficulties involved in obtaining sufficiently accurate Brinell hardness figures for cast iron, in my view, preclude the use of this method in this connection. This appears to be recognized, as one reason that is advanced for the scatter of the tensile strength-hardness data lies in the fact that there is no uniformity in the place the hardness readings were taken.

However, this difficulty would be in a large measure removed if it were possible to standardize some method of taking Brinell readings, and I think your suggestion that action be taken in this direction should be supported.

In my opinion this very detailed study is very useful and definitely worthwhile.

MR. J. T. MACKENZIE (*author*).—Mr. Massari made a point that interested me. I must confess I never thought of making a weak part that would break before something else would. In regard to the point raised by Mr. Vanick, we have to standardize for the particular purpose of specification and anybody who wants a surface hardness could not get it with the Brinell instrument anyhow because a flat must be ground.

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[4] 1944 Book of A.S.T.M. Standards, Part I, p. 634
[5] Doctor of Metallurgy, Ashleigh, England.

COMPRESSIVE STRESS-STRAIN PROPERTIES OF SOME AIRCRAFT MATERIALS*

By P. E. Sandorff[1] and R. K. Dillon[1]

Synopsis

A comprehensive investigation was made to establish compression stress-strain data and tangent-modulus *versus* stress data for the commonly used aircraft materials. The test fixtures and extensometer equipment developed for this work are rugged, accurate, and rapid in operation. Their construction and use are described, and methods of analyzing and presenting the data are discussed. Curves are presented which show the stress-strain characteristics for materials as tested as well as for those which would have specified minimum strength properties.

Each different structural material, which is made according to certain specifications regarding composition, manufacturing and processing, is recognized as having a particular set of mechanical properties (tensile strength, tensile and compressive yield stress, modulus, elongation, etc.). While any variation in the manufacture or treatment of the material may affect the mechanical properties, the specifications and manufacturing practices restrict such variations to a small range for any one material. By statistical study this range has been explored for commonly used materials, and minimum values acceptable under a given specification have been determined. Such "specified minimum properties" are tabulated in ANC-5 (**1**).[2]

Engineering design and research requirements also include complete definition of the stress-strain curves of the structural materials. The stress-strain curves, similar to the other mechanical properties, vary among different specimens of a particular material. Representative curves conforming to the specified minimum properties are desired. The needs are particularly strong with regard to the compressive stress-strain properties, and to that end this investigation was directed.

To accomplish this purpose, it was first necessary to develop methods and equipment by which uniformly accurate test data on the shape of the compressive stress-strain curve could be obtained speedily and economically. Secondly, a reasonable method of sampling was established and a coordinated series of tests was made, so that for each subject material of a particular designation several independent sets of test data were obtained. This gave an indication of the scatter band. Finally, for each material, each set of stress-strain data was scaled down to conform to the specified properties by affine transformation, and an average curve was drawn.

The results were also presented as curves of tangent-modulus *versus* stress for the specified minimum properties. Each of these was then expanded into a family of curves to show the variations which may occur within a particular material as a function of the yield strength. The presentation assumes that a linear affine relationship exists only between all stress-strain curves of a particular material designation.

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*Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

[1] Structures Research Laboratory, Lockheed Aircraft Corp., Burbank, Calif.

[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 1051.

SCOPE OF INVESTIGATION AND SELECTION OF SPECIMENS

The investigation was intended to fill the need for information as it became evident. The test work was begun in 1943, and to date about eighty different materials have been investigated. These include aluminum, magnesium, and steel alloys, in sheet, plate, extrusion, billet, etc., with-grain and across-grain and with various heat treatments.

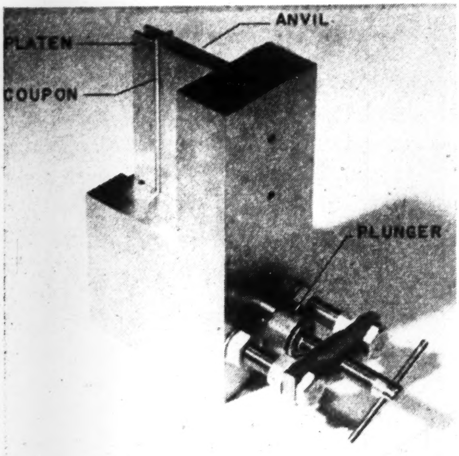

FIG. 1.—Compression Coupon Test Fixture.

In a few of these cases no more than two specimens were prepared and tested, the extent of the study being determined by practical considerations. For the most part, particularly in sheet materials, four to six coupons were obtained. These were cut from different gage material, from different locations, from different mill stock, etc., to obtain as much variation as was reasonably possible under the specifications established by each material designation.

Specimens cut from sheet stock were of the single-thickness coupon type and were tested in the fixture described below. Specimens cut from plate, extrusion, casting, etc., were usually made similar to the sheet stock coupons, the thickness being chosen as convenient for the investigation with additional smooth machined surfaces if required. Some specimens were made circular in section for simplicity of machining, or of section identical with the original extrusion or bar; these were designed for flat-end loading between the platens of the test machine with no additional support.

The determination of the stress-strain curves was extended much further into the plastic region than is generally done in such tests. In many laboratories the 0.2 per cent offset yield point is regarded as the practical outer limit to the plastic range. While the first portion of the curve is the most important, the range of interest is much broader. Extreme fiber strains in most aircraft structural elements will greatly exceed the yield point before failure. Some structural studies require accurate definition of the plastic range in compression to at least 0.5 per cent offset, and it appears reasonable to demand that the data, in any case, be complete enough to plot a tangent-modulus column curve to a slenderness ratio of 10.

TEST METHODS AND EQUIPMENT

Until recently, accurate compressive stress-strain data of the type desired were difficult and costly to obtain. This was particularly true of thin sheet materials, which in the present instance were of major interest. Significant advancements in methods and equipment, which may be traced in recent literature, (**2–12**) have largely overcome these difficulties. Many of the referenced items strongly influenced the designs described below.

Compression Coupon Test Fixture:

The test apparatus used for thin sheet coupons provides restraint against lateral buckling by means of hardened and smooth-ground steel platens, which bear against the faces of the coupon through a thin film of lubricant. This method is similar to that developed at the Bureau of Standards (**11**) and used widely in the industry. In the design of the Lockheed equipment attention was given to ease of handling, accessibility for mounting the extensometer, provision for applying any desired lateral restraining pressure to the faces of the coupon, and flexibility in accommodating various size specimens.

The test fixture and its constructional details are shown in Figs. 1 and 2. Essentially a soft spring is used to apply a lateral restraining pressure to the coupon, similar in principle to Van den Broek's arrangement (**9**). This contrasts with the arrangement used by Miller (**11**) and others, (**6, 8, 10**) in which lateral guides are located with considerable rigidity. With the latter arrangement the strain at which local buckling occurs and the wavelength of the local buckling depends (exclusive of coupon properties) on the gap or film thickness of lubricant remaining between the restraining guides and the surfaces of the specimen—usually a matter of tenths of thousandths of an inch—and this gap must be adjusted by "feel." The gap is also reduced by lateral expansion of the specimen as plastic deformation becomes appreciable, and the frictional errors may then increase. The control of lateral restraining pressure, on the other hand, is a relatively easy matter and insures trouble-free exploration far into the plastic range. The buckling strain in this case depends on the pressure applied normal to the surfaces and on the spring constant. A reasonable combination is readily found; for example, with the equipment described a confining load of 75 lb. on a coupon 0.064 by $\frac{1}{2}$ by 3 in. of 24S-T86 material will permit determination of the stress-strain curve to 0.016 compressive strain. Under this pressure

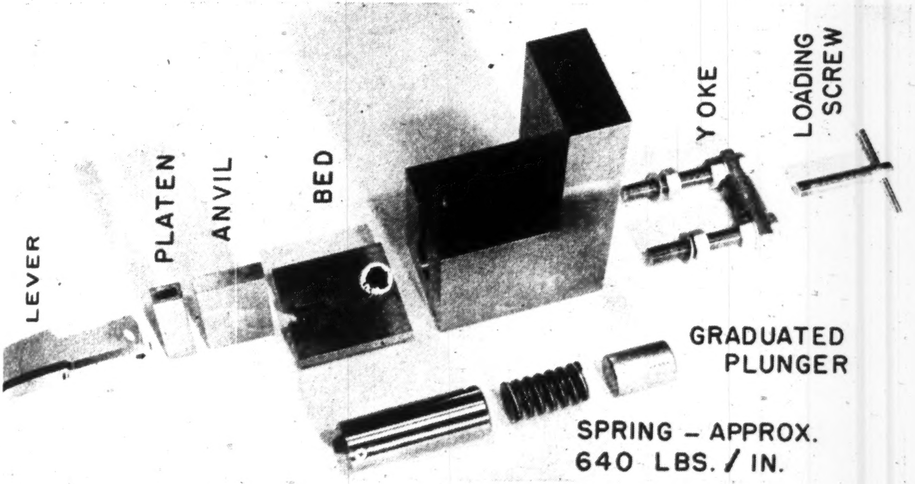

FIG. 2.—Compression Coupon Test Fixture Disassembled.

and with proper lubricant the frictional error is less than 2 lb. Choice of lubricant is important, a heavy fiber grease such as that recommended by Miller (11) being superior to all others tried, except possibly a specially developed paraffin and oil combination.

The test fixture finds universal applica-

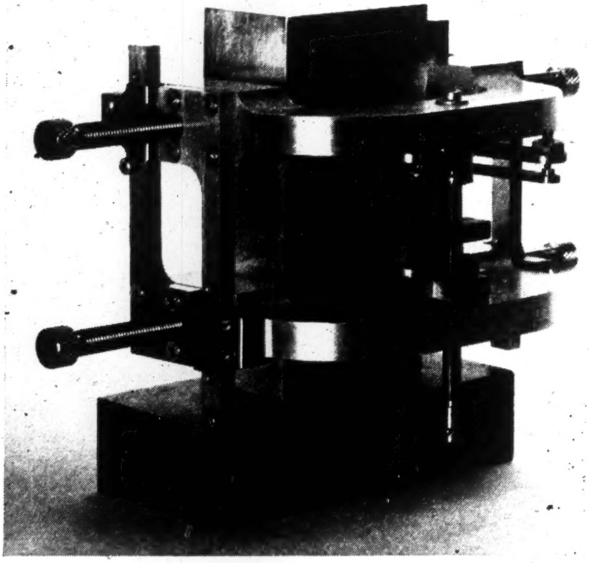

FIG. 3.—Lockheed Extensometer (Averaging Type) Mounted on 1 by 2 by 4-in. Compression Specimen.

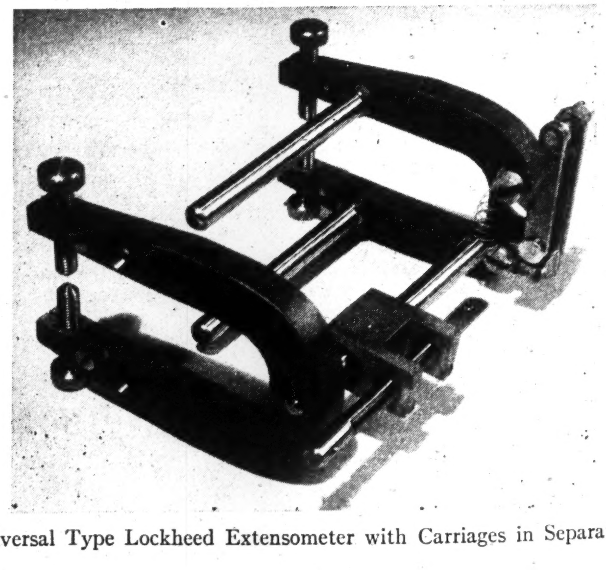

FIG. 4.—Universal Type Lockheed Extensometer with Carriages in Separated Position.

tion in that a variety of sizes and shapes of anvils and platens are readily made, to provide necessary lateral restraint not only to any size flat coupon but also, for example, to the flat elements of hat sections or stiffener shapes. Coupons of thickness from 0.010 to 0.312 in. have been tested with equal success.

Lockheed Extensometer:

The extensometer unit used for these tests is of a special construction designed to be used with the Templin-type automatic recording equipment (13). This equipment is available with Southwark-Emery testing machines.

The Templin equipment, with the standard extensometer units, is a highly satisfactory instrument, being rugged, fast in operation, easy to use and flexible in application, and producing data of excellent accuracy for ordinary purposes. In skilled hands the accuracy and consistency of the results are sometimes surprisingly good (7). For the purposes of the tests at hand, however, it was practicable to build a high precision extensometer unit differing in detail but using the same automatic indicating principles as the standard unit.

Two versions of the new unit are illustrated in Figs. 3 and 4. Essentially, the extensometer unit comprises two yokes or carriages, in each of which a pair of hardened steel gage points is rigidly mounted. The carriages are positioned with respect to each other so that as the specimen strains, their relative displacement can be measured by a precision-lapped micrometer screw. This screw is driven through a flexible shaft by the selsyn motor of the standard Templin equipment, and it follows the deformation automatically by making and breaking the same electrical circuit. However, the gap at the contact is magnified by a lever arrangement, greatly increasing the sensitivity yet preserving "null-point" indication.

High accuracy is obtained because, first, any small unevenness in stress over the specimen section is averaged out. Secondly, the carriage suspensions are so arranged that as the specimen strains, pure translational motion occurs with a minimum of friction. Therefore, gage-point loads remain low and deformation of the extensometer members is negligible. Finally, the size and direction of the loads on the gage points remain virtually unchanged regardless of the extent of the deflection.

The extensometer shown in Fig. 3 was designed primarily to be mounted on the edges of thin sheet compression coupons. There is therefore only one degree of freedom which must be restrained in each carriage: that of rotation about the axis of the gage points which it carries. This restraint is achieved by cantilever members which extend to the other carriage, where they may slide on a ball seat which positions them accurately against the undesired rotation. Any additional constraints are unnecessary and may introduce binding and excessive gage-point loads.

The extensometer shown in Fig. 4 is an earlier design with which a large portion of the compressive stress-strain data was obtained. It was made primarily for tension coupon tests and the system of cantilever restraining members was designed to permit only pure translation of the carriage, regardless of restraints introduced at the gage points. Therefore, when used on the edges of compression coupons, it is not a true "averaging" type extensometer; however, the drawback can be compensated by controlling other features of the test arrangement more accurately.

The sensitivity of these extensometer units, when used over a 2-in. gage length, is about ±0.00003 units of strain; the accuracy, for the same gage length, is about ±0.00010 units of strain. Their ruggedness and dependability are testified by the fact that the instrument shown in Fig. 4 is now in its fourth year of continual service. It has been used for all types of tests: tension and compression, joint specimens of all description, over gage lengths of from 1 to 30 in., and even in extreme temperature tests. In many tests it remains mounted on the specimen through destruction, providing a complete record up to fracture.

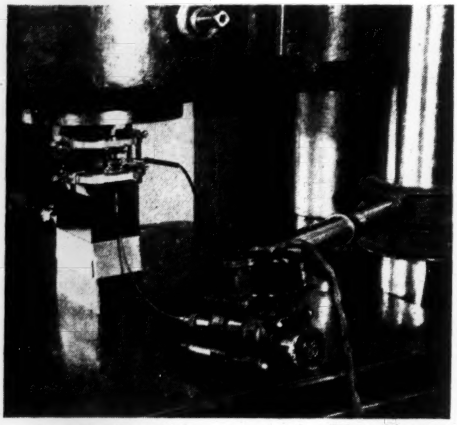

Fig. 5.—Test Arrangement for Thin Sheet Coupon.

Test Procedure:

Tests were conducted in a Southwark-Emery hydraulically actuated testing machine. Thin-stock coupons were smooth-machined square and parallel to $\frac{1}{2}$ in. and either 3.10 or 4.12 in. long. The shorter length is necessary in high-strength specimens because of the possibility of column action occurring in the plane of the sheet. The faces of each coupon were hand polished with oil and crocus cloth held on a flat surface, then cleaned and coated heavily with lubricant before inserting in the coupon test fixture. The extensometer was positioned by eye, the complete test arrangement appearing as in Fig. 5. Rate of straining was held constant throughout each test at about 0.006 strain per minute by simply closing the outflow valve and leaving the inflow valve undisturbed at a setting determined by previous experiment.

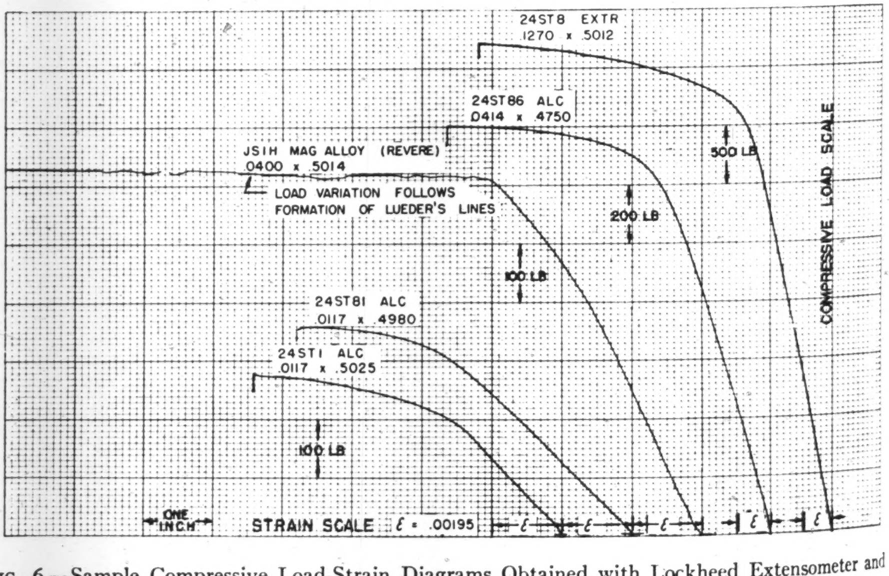

Fig. 6.—Sample Compressive Load-Strain Diagrams Obtained with Lockheed Extensometer and Thin Sheet Coupon Testing Apparatus, Recorded Automatically with Templin Equipment.

Specimens of large stable section were simply placed between parallel test platens. In any case the type of data obtained was similar. A photograph showing the type of recordings obtained is presented in Fig. 6, with explanatory notes added. Curves are shown for 0.010 gage aged and unaged 24S-T alloy, as well as some heavier materials; high values of strain were obtained before buckling or lateral bowing interfered with the test data. Of interest is the type of data obtained for some magnesium alloys which show marked Lueder's lines formations at the compression yield stress. This phenomenon is previously noted by Moore and McDonald (14). Figure 7 shows a photograph of such a coupon after test, the regularly spaced Lueder's lines being evident over the full length of the coupon.

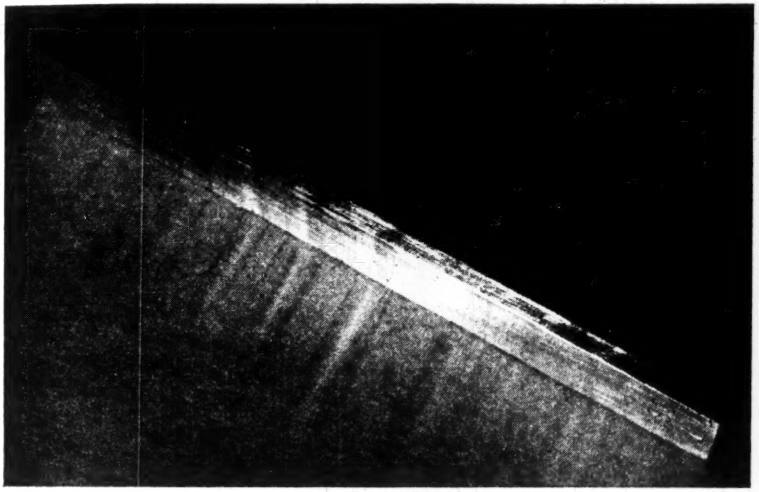

FIG. 7.—Magnesium Alloy Compression Coupon Showing Lueder's Lines Formation after Testing.

ANALYSIS OF TEST DATA

Specified Minimum Properties:

ANC-5 (1) specifies certain minimum values of the mechanical properties of various materials, for design purposes. The modulus of elasticity and the 0.002 offset compressive yield stress are used as a basis in establishing the corresponding compressive stress-strain curves.

For aluminum alloys the proportional limit is seldom specified. The method of reduction used is therefore in accord with the other properties and with the shape of the test curve. The test proportional limit is then reduced in the same ratio as the compressive yield stress.

The yield strain, corresponding to the specified compressive yield stress, is used in the analysis of test data and may be calculated as follows:

$$e_y = \frac{F_y}{E} + 0.002$$

where:

e_y = specified yield strain in inches per inch,

F_y = specified yield stress in pounds per square inch,

E = modulus of elasticity, and

0.002 = permanent strain corresponding to 0.2 per cent offset yield point.

Affine Transformation of Test Curve:

To reduce the test stress-strain curve

so that the shape is preserved while the properties correspond to specified modulus and yield strength, an affine transformation is used. Similar mathematical methods have been applied previously to all aluminum alloys as one class or to all S.A.E. X4130 steel alloys as one class (**15**), and to express stress-strain curves mathematically (**16**, **12**).

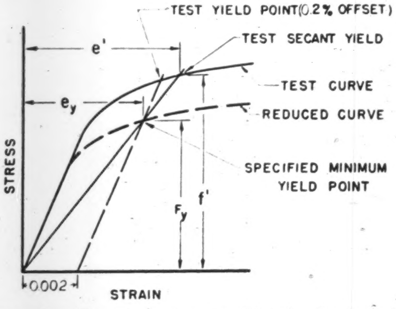
TEST YIELD POINT(0.2% OFFSET)
TEST SECANT YIELD
e'
e_y
TEST CURVE
REDUCED CURVE
SPECIFIED MINIMUM YIELD POINT
STRESS
F_y
f'
0.002
STRAIN

FIG. 8.—Construction to Obtain Reduced Stress-Strain Curve from Test Curve.

In making an affine transformation it is assumed that one curve may be obtained from another by applying a constant scale factor to each coordinate. Thus if a curve is defined by the series of points (x_1, y_1), (x_2, y_2); etc., and the chosen scale factors and K_x and K_y, the points defining the second curve would be $(K_x x_1, K_y y_1)$; $(K_x x_2, K_y y_2)$; etc.

The construction is illustrated in Fig. 8. The specified minimum yield point is given by the yield stress F_y and the yield strain e_y. Through this point a secant is drawn, intersecting the test curve at the point (f', e'). In the usual case, the modulus of elasticity of the test curve is practically identical to the specified value, and the point (f', e') lies above the 0.2 per cent offset yield stress of the test curve. The ratios of the values f' and e' to the coordinates of the specified minimum yield point determine the scale factors for the transformation.

The constant scale factor for the stress values is:

$$K_f = \frac{F_y}{f'}$$

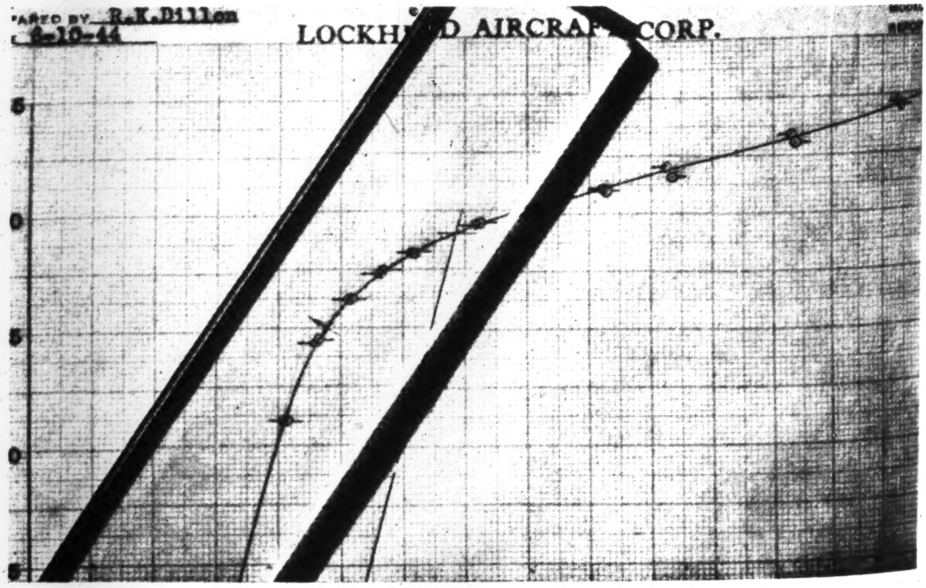
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FIG. 9.—Use of the Tangent Meter.

and the constant scale factor for strain values is

$$K_e = \frac{e_y}{e'}$$

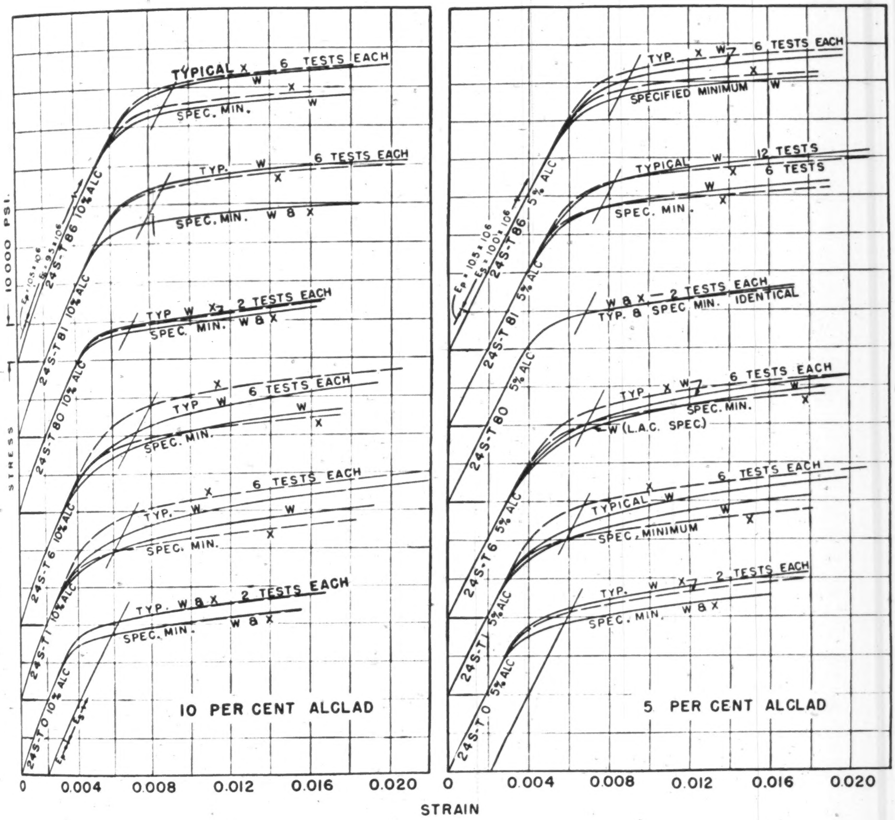

Fig. 10.—Compressive Stress-Strain Curves for 24S Alclad AN-A-13 Sheet Materials.

Specified minimum values correspond to ANC-5 revision of October, 1943, except for 24S-T6, 5 per cent alclad, for which a minimum value used by Lockheed Aircraft Corp. is shown. For each case, the typical curve represents an average of the several tests. Gages 0.032, 0.040, and 0.051 tested for 10 per cent alclad material: 0.064, 0.071, 0.081, and 0.102 for 5 per cent alclad material. All tests were run in May, 1944, on material manufactured by the Aluminum Company of America.

24S-T0 = 24S-T AN-A-13 heat-treated by user, no pre-stretch
24S-T6 = 24S-RT AN-A-13

By application of the scale factors to a sufficient number of points on the test curve, the reduced curve is accurately defined.

The scale factors K_f and K_e are equal to each other if the initial or straight line portion of the test curve is equal to the specified modulus of elasticity. Deviations of the recorded moduli are usually less than ±2 per cent and are probably due to testing technique and instrumentation (17). In this event the deviation may not be constant over the entire plastic range. The good agreement among reduced test data from different specimens of the same material indicates that such errors are small.

Curves of Stress versus Strain:

In reducing test data such as shown in Fig. 6 to obtain the stress-strain curve for specified minimum properties, the conversion from load to stress is included in the scale factor K_f. Reduced points obtained from all different specimens of the same material are plotted on one figure, and through these points a mean curve is drawn. Stress-strain curves, and the points from which they were drawn, are presented in the accompanying figures for several of the materials investigated.

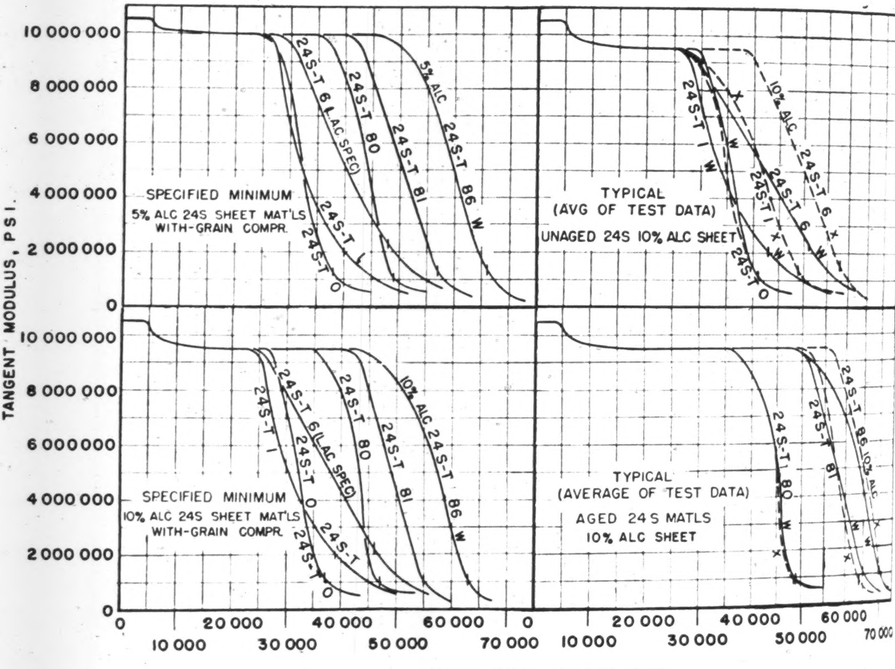

FIG. 11.—Tangent Modulus *versus* Compressive Stress Curves for Alclad 24S Sheet Materials.

"Specified minimum" curves are determined from stress-strain curves.
"Typical" curves are obtained by expanding "specified-minimum" abscissae in ratio of average test yield stress to specified minimum.—this corresponds to linear affine expansion of stress-strain curves.

Tangent Modulus versus Stress Curves:

Tangent modulus *versus* compressive stress data were obtained from the reduced stress-strain curves by means of the tangent meter shown in Fig. 9. This device consists of a lucite block upon which a fine line of short length (in this case about one-eighth inch long) is scribed parallel to one edge. Another index mark is scribed perpendicular to and crossing the first line at its center. To use the instrument, the short line is superimposed on the stress-strain curve so that it appears to replace a portion of the curve. The slope of the edge of the block is the tangent modulus at the stress indicated by the index mark. The values so determined are only approximate, but the accuracy and agreement between repeated readings are much better than those obtained by other graphical methods, mirror devices, or even analytical methods (which involve reading points off the curve).

The tangent modulus-stress data for

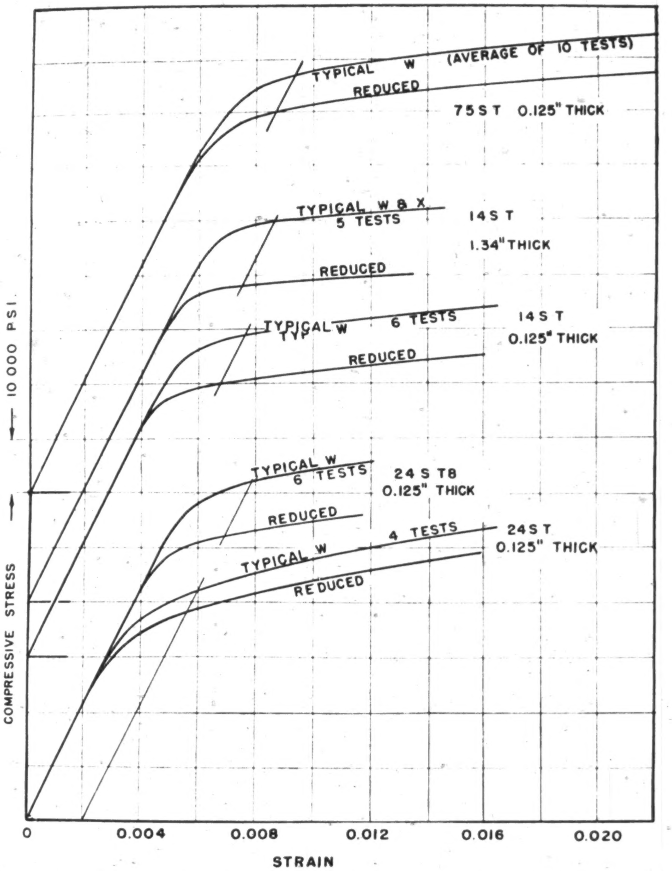

Fig. 12.—Compressive Stress-Strain Curves for Extruded Materials.

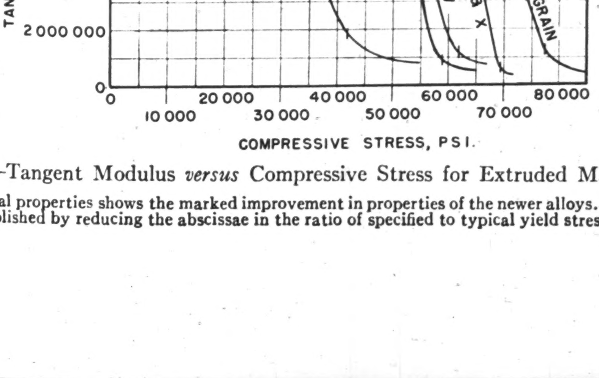

Fig. 13.—Tangent Modulus *versus* Compressive Stress for Extruded Materials.

Comparison of typical properties shows the marked improvement in properties of the newer alloys. The transformation of any curve is accomplished by reducing the abscissae in the ratio of specified to typical yield stress.

various materials are also presented in Figs. 10, 11, 12, and 13. These curves may be expanded or reduced by simply multiplying the given abscissae by the ratio of the yield stress of the desired curve to the given yield stress. This operation corresponds to a linear-affine transformation of the stress-strain curve.

As a check on the accuracy of the entire process, tangent *versus* load values were determined for one set of data directly from the test load-strain curve. These data were then converted to tangent modulus *versus* stress and transformed, by application of the ratio of yield strengths, to define the curve for specified minimum properties. The points so obtained were found to agree to within less than one per cent with the tangent modulus curve determined for the mean, reduced stress-strain curve.

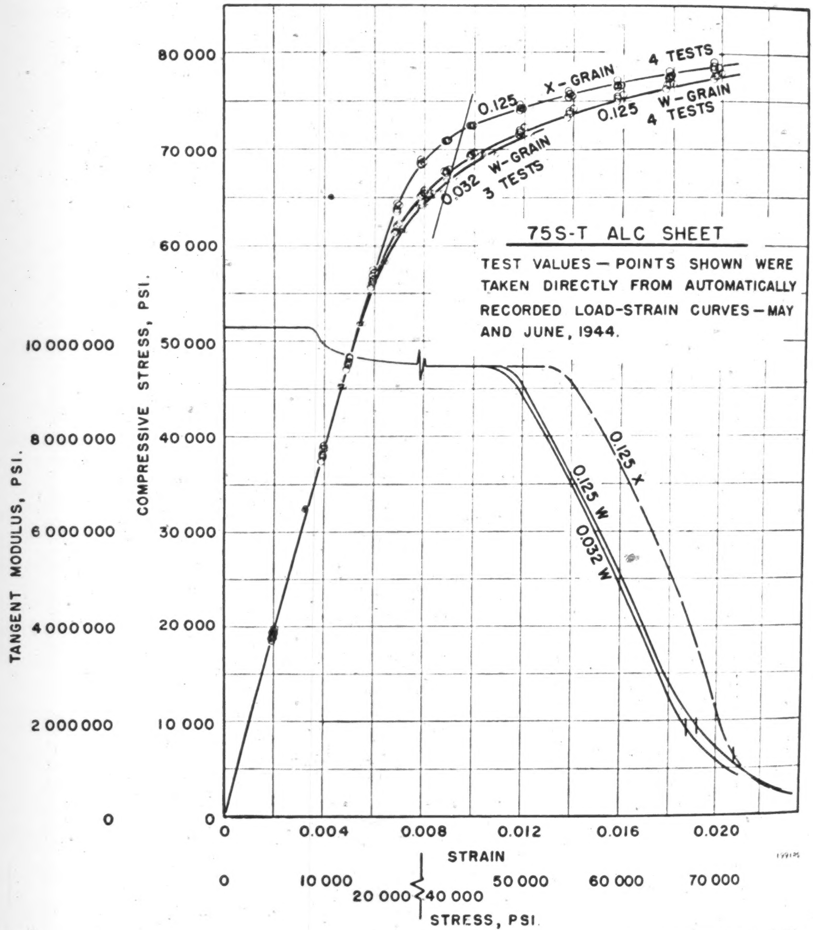

FIG. 14.—Compressive Stress-Strain and Tangent-Modulus Curves for 75S-T Alclad Sheet.

Test values—points shown were taken directly from automatically recorded load-strain curves in May and June, 1944.

Special Case of Alclad Sheet (*Fig. 14*):

Clad aluminum alloys present a special problem. The common 10 per cent alclad coating, for instance, may be considered as approximately a 2S alloy having an initial elastic modulus of 10.0 × 10^6 psi. and a proportional limit of 3000 or 4000 psi. The basic alloy may be 24S-T, which has an initial modulus of 10.6 × 10^6 psi. and a proportional limit of 25,000 psi. Simple summation of the action of the two materials indicates the behavior of the clad alloy under direct compression: the composite material has an initial modulus of 10.55 × 10^6 psi. which holds true to the proportional limit of the cladding. A plot of the tangent modulus *versus* stress curve would show at this point a sudden drop, then an asymptotic approach to the value of 9.55 × 10^6 psi.—the elastic rigidity contributed by the base alloy alone (90 per cent of 10.6 × 10^6 psi.). The shape of this portion of the tangent modulus *versus* stress curve would reflect to tenth scale the shape of the curve for the cladding material alone. Under increasing compression the effect of the cladding would become negligible at about 15,000 psi. and the material would then appear to behave just as the base alloy alone, allowing for the fact that only 90 per cent of the gross area is active.

The exact effects are difficult to determine by test, and they vary considerably with processing. To simplify analysis, it was assumed that for alclad 2S clad) materials, the primary modulus applied up to 10,000 psi., at which point the stress-strain curve breaks sharply into the secondary modulus. The yield strain may then be calculated as

$$e_y = \frac{f_{yc}}{E_p} + \frac{F_y - F_{yc}}{E_s} + 0.002$$

where:

f_{yc} = yield stress of cladding (assumed 10,000 psi.),
E_p = primary modulus of elasticity, and
E_s = secondary modulus of elasticity.

The determination of K_f and K_e and the remainder of the presentation is unchanged.

CONCLUSIONS

Compressive stress-strain and tangent-modulus *versus* stress curves conforming to minimum specified properties have been presented for materials commonly used in aircraft structure. The shape of each curve is established by test data from several representative samples.

The equipment and methods employed in this investigation constitute a practical and economical means of obtaining complete and accurate compressive stress-strain data for both thin-sheet and stable sections.

Acknowledgment:

The authors express their appreciation to Samuel Taubman, S 1/C, U. S. Naval Reserve, for his excellent work in the detail design of much of the test equipment and instrumentation.

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EVALUATION OF THE FORMING PROPERTIES IN BENDING OF FIVE COMMERCIAL SHEET ALUMINUM ALLOYS*

By G. R. Gohn[1] and S. M. Arnold[1]

Synopsis

This paper describes two methods of test developed for evaluating the forming characteristics in bending of sheet and strip metals, one by means of a punch and die setup, the other by means of a bending brake. Minimum safe forming radii are given for forming 90-deg. bends in five commercial aluminum alloys in sheet form, varying in thickness from 0.012 to 0.128 in. These alloys include 2S-$\frac{1}{2}$H, 3S-$\frac{1}{2}$H, 24S-O, 24S-T, 52S-$\frac{1}{4}$H, 52S-$\frac{1}{2}$H and 61S-T. The data include minimum radii suitable for forming 90-deg. bends in these materials on punch-press setups and on bending brakes.

In the manufacture of telephone apparatus, many contact springs and structural parts are used which have been formed from thin sheets of nonferrous metal in a series of blanking, punching, and forming operations. When the number of parts involved is large, the work is generally done on punch and die setups; when the number of parts involved is small, the work may be done on shears and bending brakes. Parts bent over too sharp a radius will crack, and parts bent over too large a radius may require excessive space. Hence it is desirable, from a design and manufacturing standpoint, to have data available relative to the minimum forming properties of the various sheet metals in the range of thicknesses and tempers frequently used. These may vary from 0.012 to $\frac{3}{8}$ in. in thickness and from full anneal to spring temper.

Considerable data on the punch-press forming properties of brass, phosphor bronze, and nickel-silver strip, as well as a limited amount of data on some of the other copper-base alloys and on some aluminum alloys in sheet form are given in a paper by Gohn.[2] Additional data on some of these same alloys may be found in a paper by Straw, Helfrick, and Fischrupp.[3] While there are, at the present time, no suitable data available on the brake-forming characteristics of thin sheet metal, the authors have been informed that reports covering fundamental studies in this field have been issued by the National Research Committee of the Office of Scientific Research and Development, War Metallurgy Division. These reports, however, had not been declassified at the time this paper was prepared and hence the authors have had no opportunity to review the technical data contained therein.

In recent years, with the increase in the amount of war work, there has been a marked increase in the number of formed aluminum parts used in the manufacture of chasses, cabinets, and other structural parts where the quan-

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[1] Members of Technical Staff, Bell Telephone Laboratories, Inc., New York, N. Y.

[2] G. R. Gohn, "The Forming Properties of Some Non-Ferrous Sheet Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 207 (1936).

[3] W. A. Straw, M. D. Helfrick, and C. R. Fischrupp, "Forming Properties of Thin Sheets of Some Non-Ferrous Metals," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Inst. Metals Div., p. 317 (1931).

tities have been small. While some of this work was done on punch and die setups, much of the work, because of the limited number of parts involved, was done by hand or on bending brakes. Consequently a need arose for more complete forming data on the commonly used aluminum alloys, particularly as applied to bending brakes. There was also some evidence that the forming characteristics of aluminum alloys, as now rolled, differed from those as determined in 1936. Therefore the punch-press forming studies were repeated on some of the aluminum alloys and the results of these tests were compared with the results of the brake-forming tests.

TESTING PROGRAM

The materials included in this investigation were commercial 2S-$\frac{1}{2}$H, 3S-$\frac{1}{2}$H, 24S-O, 24S-T, 52S-$\frac{1}{4}$H, 52S-$\frac{1}{2}$H, and 61S-T, obtained from the Whitehead Metal Products Co. of New York, N. Y. The 17S alloy listed in the previous study[2] was not included in this study because it has been largely replaced by the stronger 24S alloy of somewhat similar composition. The newer 75S alloy was not included because, at the time these tests were made, this material was not available, its use being restricted to aircraft applications. The materials tested ranged in thickness from 0.012 to 0.128 in.

METHODS OF TEST

The test specimens used in this study were $\frac{3}{4}$ by 3-in. sheared specimens taken parallel, at 45 deg., and at 90 deg. to the direction of rolling, from sheets of each of the five aluminum alloys. In the case of the heavier thicknesses, it was necessary to mill the 3-in. edges of the specimens to obtain samples sufficiently free from shearing stresses to permit a true evaluation of the basic forming characteristics.

In determining the punch-press forming characteristics, a series of 90-deg. punches (Fig. 1) having sharp, $\frac{1}{64}$, $\frac{1}{32}$, $\frac{3}{64}$, $\frac{1}{16}$, $\frac{3}{32}$, $\frac{1}{8}$, $\frac{5}{32}$, $\frac{3}{16}$, $\frac{7}{32}$, $\frac{1}{4}$, and $\frac{3}{8}$-in. radii, were used in a punch-and-die setup as shown in Fig. 2. Figure 1 also shows punches having $\frac{1}{2}$, $\frac{3}{4}$, and 1-in. radii which were used in the previous investigation but were not required for these studies. To determine the forming characteristics of a material the punch and die (Fig. 2) were set up and the distance between the punch and die at the end of the downward stroke adjusted to the approximate thickness of the test specimen. The specimen was then placed unclamped on the die which has a sharp, 90-deg. V-groove and the specimen formed in the usual manner on the punch press. For this work a Bliss No. 18 punch press was used.

A group of five specimens was formed over a punch of a given radius and these specimens were examined visually by comparison with a series of standard specimens, such as that shown in Fig. 3, to determine whether the forming radius was satisfactory. Parts which are formed over too sharp a radius will crack, as illustrated by specimens Nos. 1 to 4 in Fig. 3. As the radius is increased the cracking becomes less and less pronounced until there is no visible open crack, but instead a pronounced "orange peel" is evident. This condition is illustrated by specimens Nos. 5 to 7 in Fig. 3. A pronounced "orange peel" is objectionable because such a part is weaker than one free from this condition, and because it detracts from the appearance of the finished part. Therefore, increasingly larger radii were used until the "orange peel" effect became less marked, as illustrated by specimens Nos. 8 to 10 in Fig. 3. The

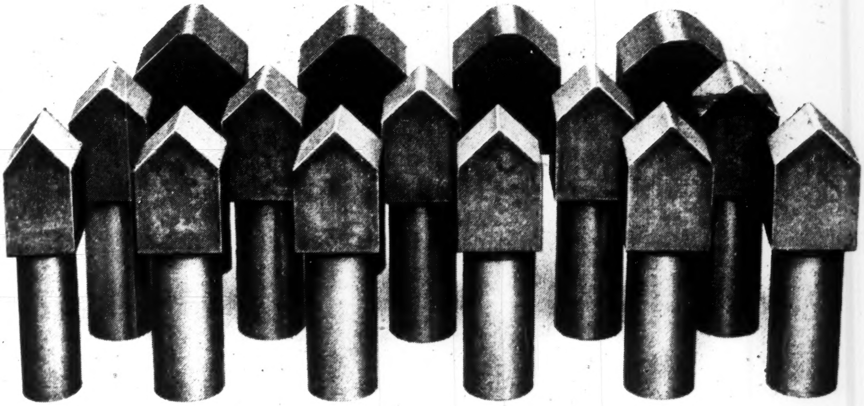

FIG. 1.—Punches.

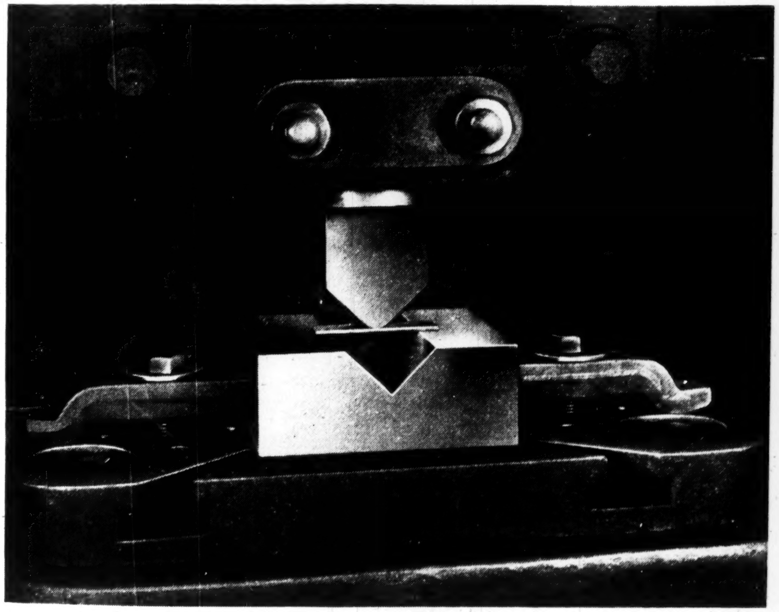

FIG. 2.—Punch and Die Setup in Punch Press.

surface shown on specimen No. 8 in Fig. 3 (also illustrated by the insert) is considered satisfactory and the radius used in forming this specimen is considered to be the minimum satisfactory radius for the material tested.

For the brake-forming studies a 12-in. Di-Acro bending brake was modified as shown in Fig. 4. This modification consisted of attaching to the fixed platen of the brake two supporting members containing 45-deg. slots. A slide moves

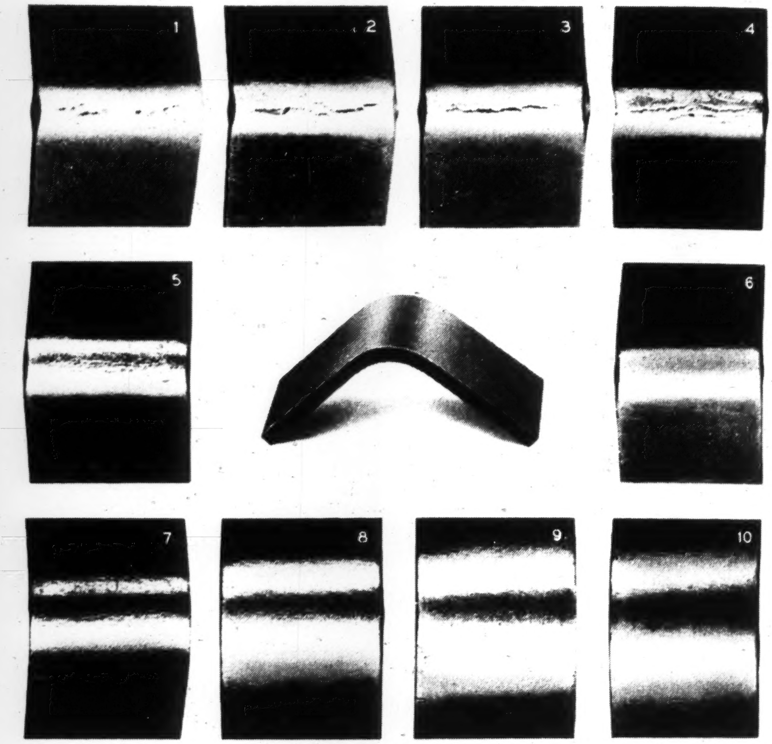

FIG. 3.—Sections of Formed Specimens (Insert—Satisfactorily Formed Specimen).

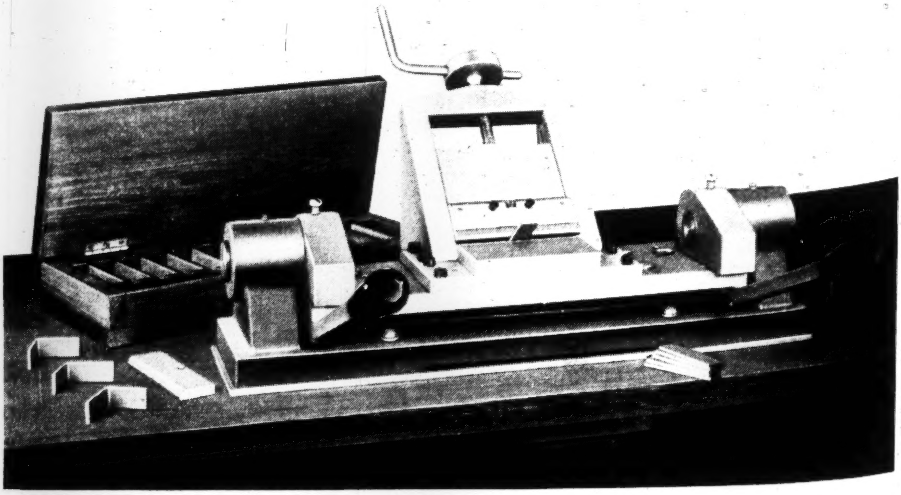

FIG. 4.—Modified Bending Brake.

up and down in these slots at a 45-deg. angle to the fixed platen. This slide is controlled by a screw by means of which clamping pressure can be exerted upon the specimen. To the slide are attached removable mandrels of various radii. These mandrels, shown in Fig. 4, have sharp, $\frac{1}{64}$, $\frac{1}{32}$, $\frac{3}{64}$, $\frac{1}{16}$, $\frac{3}{32}$, $\frac{1}{8}$, $\frac{5}{32}$, $\frac{3}{16}$, $\frac{7}{32}$, $\frac{1}{4}$, and $\frac{3}{8}$-in. radii.

The radius mandrel, when mounted on the movable slide, is so located that a plane passed through the axis of rotation of the movable platen and normal to the surface of the fixed platen is tangent to the mandrel radius when the slide is lowered against the fixed platen. Since the slide moves at an angle of 45 deg. to the fixed platen, the mandrel moves away from the normal plane through the axis of rotation by an amount equal to the thickness of the specimen under test. Consequently there is no necessity for adjusting the horizontal position of the mandrel for variations in thickness of stock. This is in marked contrast to the punch and die setup which requires adjustment for each thickness, and to the commercial bending brakes which require adjustment for each change in mandrel radius and thickness of the stock.

In use, the slide and the attached mandrel are raised by means of the screw handle, a specimen is inserted beneath the mandrel and clamped by tightening this handle. Once the specimen is clamped, it is formed around the mandrel by rotating the movable platen through an angle of 90 deg. The specimen is then unclamped and examined visually. As in the case of the punch-press studies, groups of five specimens were formed over successively larger radii until satisfactory forming was obtained as previously described.

The tensile strength of each of the sheets of aluminum used in these studies was determined on test specimens taken parallel to the direction of rolling, in accordance with standard laboratory methods. Test specimens similar to those shown in Fig. 2 of A.S.T.M. Standard Methods of Tension Testing of Metallic Materials (E 8 – 42)[4] were used. The width and thickness were measured to the nearest 0.0001 in. The specimens were tested on a 20,000-lb. Amsler universal testing machine at a maximum free head speed of 0.100 in. per in. of length. The testing machine was accurate to ± 1 per cent at all load readings. The scale used was such that fracture of the test

TABLE I.—CHEMICAL COMPOSITION.

	Alloy 2S	Alloy 3S	Alloy 24S	Alloy 52S	Alloy 61S
Specification	QQ-A-561[a]	QQ-A-359a[a]	AN-A-12[b]	QQ-A-318[a]	QQ-A-327[a]
Composition, per cent:					
Aluminum	99.00 min.	remainder	remainder	remainder	remainder
Manganese	...	1.0 to 1.5	0.3 to 0.9	0.10 max.	0.15 max.
Copper	...	0.2 max.	3.8 to 4.9	0.10 max.	0.15 to 0.4
Iron	...	0.7 max.	0.5 max.	...	0.7 max.
Silicon	...	0.6 max.	0.5 max.	...	0.4 to 0.8
Zinc	...	0.1 max.	0.1 max.	0.03 max.	0.2 max.
Chromium	...	...	0.25 max.	0.15 to 0.35	0.35 max.
Magnesium	...	...	1.2 to 1.8	2.2 to 2.8	0.8 to 1.2
Lead	...	...	...	...	...
Titanium	...	...	...	...	0.15 max.
Other elements:					
Each	...	0.05 max.	0.05 max.	0.03 max.	0.03 max.
Total	...	0.15 max.	0.15 max.	0.10 max.	0.10 max.

[a] Federal Specification.
[b] Army-Navy Aeronautical Specification.

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[4] 1944 Book of A.S.T.M. Standards, Part I, p. 962.

TABLE II.—MINIMUM RADII SUITABLE FOR FORMING 90-DEG. BENDS IN ALUMINUM ALLOY SHEET

ALUMINUM ALLOY 2S - ½H.

Equivalent Specifications.—A.S.T.M. Tentative Specification B 25, Federal Specification QQ-A-561, Navy Department Specification 47A2, S.A.E. Specification 25.

Designation	Nominal Thickness, in.	Actual Thickness, in.	Tensile Strength, psi.	Minimum Suitable Radius, in.						Required Bending Radii as Listed in 1944 Alcoa Handbook[a]
				For Brake Forming			For Punch Press Forming			
				Bend Perpendicular to Direction of Rolling	Bend at 45 deg. to Direction of Rolling	Bend Parallel to Direction of Rolling	Bend Perpendicular to Direction of Rolling	Bend at 45 deg. to Direction of Rolling	Bend Parallel to Direction of Rolling	Grain Direction Not Specified
....	All thicknesses	...	16 000 min. (req'd.)	...	...	...	...	...	...	...
A30......	0.012	0.0125	17 200	Sharp	Sharp	Sharp	Sharp	Sharp	Sharp	...
A1.......	0.014	0.014	18 300	Sharp	Sharp	Sharp	Sharp	Sharp	Sharp	...
A2.......	0.016	0.016	17 900	Sharp	Sharp	1/64	Sharp	Sharp	Sharp	0
A28......	0.020	0.021	17 900	Sharp	1/64	1/64	Sharp	Sharp	Sharp	...
A3.......	0.025	0.026	17 500	1/64	1/64	1/64	Sharp	Sharp	1/64	...
A4.......	0.032	0.032	18 100	1/64	1/64	1/64	Sharp	1/64	1/64	0
A31......	0.040	0.042	18 400	1/64	1/64	1/32	1/64[b]	1/64[b]	1/64	...
A32......	0.051	0.051	17 100	1/64	1/64	1/32	1/64[b]	1/64[b]	1/64	...
A5.......	0.064	0.064	16 800	1/64	1/64	1/32	1/64[b]	1/64[b]	1/64	0
....	0.072	...	...	1/64[c]	1/64[c]	1/32[c]	1/64[c]	1/64[c]	1/32[c]	...
A29......	0.081	0.083	17 400	1/64	1/64	1/32[d]	1/64	1/64	1/32[d]	...
A6.......	0.091	0.093	16 900	1/64	1/64	1/32[d]	1/64[b]	1/64[b]	1/32[d]	...
A46......	0.102	0.101	16 500	1/64	1/64	1/32	1/64[b]	1/64[b]	1/32[d]	...
A7.......	0.128	0.123	17 100	1/32	1/32	1/32	1/64	1/64	1/32	0

ALUMINUM ALLOY 3S - ½H.

Equivalent Specifications.—A.S.T.M. Tentative Specification B 79, Federal Specification QQ-A-359, Navy Department Specification 47A4, S.A.E. Specification 29.

Designation	Nominal Thickness, in.	Actual Thickness, in.	Tensile Strength, psi.	Brake: Bend Perpendicular	Brake: Bend at 45 deg.	Brake: Bend Parallel	Punch Press: Bend Perpendicular	Punch Press: Bend at 45 deg.	Punch Press: Bend Parallel	Grain Direction Not Specified
....	All thicknesses	...	19 500 min. (req'd.)	...	...	...	...	...	...	...
....	0.012	...	...	Sharp[c]	Sharp[c]	Sharp[c]	Sharp[c]	Sharp[c]	Sharp[c]	...
....	0.014	...	...	Sharp[c]	Sharp[c]	Sharp[c]	Sharp[c]	Sharp[c]	Sharp[c]	...
....	0.016	...	...	Sharp[c]	Sharp[c]	1/64[c]	Sharp[c]	Sharp[c]	Sharp[c]	...
A36......	0.020	0.020	22 200	1/64	1/64	1/32	Sharp	Sharp	1/64	0
A33......	0.025	0.025	21 900	1/64	1/64	1/32	Sharp	Sharp	1/64	...
A58......	0.032	0.031	23 400	1/64	1/64	1/32	Sharp	Sharp	1/64	0
A52......	0.040	0.042	23 000	1/64	1/32	1/32	1/64	1/32	1/32	...
A57......	0.051	0.050	22 500	1/64	1/32	1/32	1/64	1/32	1/32	...
A56......	0.064	0.062	22 400	1/64	1/32	1/32	1/64	1/32	1/32	0
....	0.072	...	...	1/32[c]	1/32[c]	1/32[c]	1/32[c]	1/32[c]	1/32[c]	...
A51......	0.081	0.084	22 900	1/32	1/32	1/32	1/32	1/32	1/32	...
A69......	0.091	0.092	22 000	1/32	1/32	1/32	1/32[d]	1/32	1/32	...
A56......	0.102	0.101	23 100	1/32	3/64	1/16	1/32	3/64	1/16	...
A49......	0.128	0.125	21 300	1/32	1/16	1/16	1/32	1/16	1/16	0 to 1t

ALUMINUM ALLOY 24S - O.

Equivalent Specifications.—Federal Specification QQ-A-355, Army-Navy Aeronautics Specification AN-A-12, S.A.E. Specification 24.

Designation	Nominal Thickness, in.	Actual Thickness, in.	Tensile Strength, psi.	Brake: Bend Perpendicular	Brake: Bend at 45 deg.	Brake: Bend Parallel	Punch Press: Bend Perpendicular	Punch Press: Bend at 45 deg.	Punch Press: Bend Parallel	Grain Direction Not Specified
....	All thicknesses	...	35 000 max. (req'd.)	...	...	...	...	...	...	...
A8.......	0.012	0.0124	31 000	Sharp	Sharp	Sharp	Sharp	Sharp	Sharp	...
....	0.014	...		Sharp[c]	Sharp[c]	Sharp[c]	Sharp[c]	Sharp[c]	Sharp[c]	...
A43......	0.016	0.016	26 500	Sharp	Sharp	Sharp	Sharp	Sharp	Sharp	0
A9.......	0.020	0.020	30 000	Sharp	Sharp	Sharp	Sharp	Sharp	Sharp	...
A77......	0.025	0.026	30 100	Sharp	Sharp	Sharp	Sharp	Sharp	Sharp	...
A10......	0.032	0.032	33 000	1/64	1/64	1/64	Sharp	Sharp	Sharp	0
A44......	0.040	0.040	30 000	1/64	1/64	1/64	1/64	1/64	1/64	...
A40......	0.051	0.051	33 600	1/64	1/64	1/64	1/64	1/64	1/64	...
A11......	0.064	0.066	29 400	1/64	1/64	1/64	1/64	1/64	1/64	0
A67......	0.072	0.072	28 600	1/64	1/64	1/64	1/64	1/64	1/64	...
A81......	0.081	0.081	29 900	1/64	1/64[f]	1/64	1/64	1/64	1/64[f]	...
A80......	0.091	0.092	32 600	3/64	3/64[f]	3/64	3/64	3/64	3/64[f]	...
A45......	0.102	0.104	26 400	3/64[f]	3/64[f]	3/64[f]	3/64[f]	3/64[f]	3/64[f]	...
A41......	0.128	0.128	37 000[e]	3/16	3/16	3/16	3/16	3/16	3/16	0

TABLE II.—*Continued*

Designation	Nominal Thickness, in.	Actual Thickness, in.	Tensile Strength, psi.	Minimum Suitable Radius, in.						Required Bending Radii as Listed in 1944 Alcoa Handbook[a]
				For Brake Forming			For Punch Press Forming			Grain Direction Not Specified
				Bend Perpendicular to Direction of Rolling	Bend at 45 deg. to Direction of Rolling	Bend Parallel to Direction of Rolling	Bend Perpendicular to Direction of Rolling	Bend at 45 deg. to Direction of Rolling	Bend Parallel to Direction of Rolling	
ALUMINUM ALLOY 24S - T. *Equivalent Specifications.*—Federal Specification QQ-A-355, Army-Navy Aeronautics Specification AN-A-12, S.A.E. Specification 24.										
....	All thicknesses	...	64 000 min. (req'd.)	...	...	...	...	...	...	...
....	0.012	...	...	...	...	...	...	...	...	...
....	0.014	...	...	...	...	...	...	...	...	...
....	0.016	...	...	...	...	...	...	...	...	1½-3t
A34	0.020	0.020	71 200	1/32	1/32	1/32	1/32	1/32	1/32	...
....	0.025	...	...	3/64^c	3/64^c	3/64^c	3/64^c	3/64^c	3/64^c	...
A12	0.032	0.033	72 200	1/16	1/16	1/16	1/16	1/16	1/16	2t-4t
A13	0.040	0.040	69 700	3/32	3/32	3/32	3/32	3/32	3/32	...
A14	0.051	0.049	69 000	1/8^g	1/8^g	1/8	1/8^g	1/8^g	1/8	...
A15	0.064	0.063	71 700	5/32	5/32	5/32	5/32	5/32	5/32	3t-5t
....	0.072	...	...	3/16^c	3/16^c	3/16^c	3/16^c	3/16^c	3/16^c	...
A54	0.081	0.079	71 500	7/32	7/32	7/32	7/32	7/32	7/32	...
A61	0.091	0.091	71 600	7/32	7/32	7/32	7/32	7/32	7/32	...
A55	0.102	0.100	72 000	1/4	1/4	1/4	1/4	1/4^h	1/4	...
A16	0.128	0.127	72 600	3/8	3/8	3/8	3/8	3/8	3/8	4t-6t
ALUMINUM ALLOY 52S - ¼ H. *Equivalent Specifications.*—A.S.T.M. Tentative Specification B 109, Federal Specification QQ-A-318, Navy Department Specification 47A11, S.A.E. Specification 201.										
....	All thicknesses	...	31 000 min. (req'd)	...	...	...	...	...	...	...
....	0.012	...	...	Sharp^c	Sharp^c	Sharp^c	Sharp^c	Sharp^c	Sharp^c	...
....	0.014	...	...	Sharp^c	Sharp^c	Sharp^c	Sharp^c	Sharp^c	Sharp^c	...
....	0.016	...	...	Sharp^c	Sharp^c	Sharp^c	Sharp^c	Sharp^c	Sharp^c	0
A73	0.020	0.021	33 400	1/64	1/64	1/64	1/64	1/64	1/64	...
A60	0.025	0.028	34 400^i	1/64	1/32	1/32	1/64	1/64	1/32	...
....	0.032	...	...	1/32^c	3/64^c	3/64^c	1/32^c	3/64^c	3/64^c	0
A19	0.040	0.039	32 900	1/32	3/64	3/64	1/32	3/64	3/64	...
A20	0.051	0.050	32 800	3/64	3/64	1/16	3/64	3/64	1/16	...
A21	0.064	0.064	33 600	1/16	1/16	3/32	1/16	1/16	3/32	0
....	0.072	...	...	3/32^c	3/32^c	1/8^c	3/32	3/32^c	1/8^c	...
A22	0.081	0.081	33 800	3/32	3/32	1/8	3/32	3/32	1/8	...
A23	0.091	0.090	33 100	1/8	1/8	1/8	1/8	1/8	1/8	...
....	0.102	...	...	1/8^c	1/8^c	1/8^c	1/8^c	1/8^c	1/8^c	...
A24	0.128	0.123	34 100^j	1/8	1/8	1/8	1/8	1/8	1/8	0-1t
ALUMINUM ALLOY 52S-½H. *Equivalent Specifications.*—A.S.T.M. Tentative Specification B 109, Federal Specification QQ-A-318, Navy Department Specification 47A11, S.A.E. Specification 201.										
....	All thicknesses	...	34 000 min. (req'd.)	...	...	...	...	...	...	...
....	0.012	...	...	...	...	...	...	...	...	...
....	0.014	...	...	...	...	...	...	...	...	...
....	0.016	...	...	...	...	...	...	...	...	0
....	0.020	...	...	...	...	...	...	...	...	...
A48	0.025	0.027	36 400	1/32	1/32	3/64	1/32	1/32	1/32	...
A39	0.032	0.033	36 300	3/64	3/64	3/64	3/64	3/64	3/64	0
A35	0.040	0.042	35 500	3/64	3/64	1/16	3/64	3/64	1/16	...
A40	0.051	0.051	33 600^k	3/64	3/64	1/16	3/64	3/64	1/16	...
A25	0.064	0.063	35 000	1/16	1/16	3/32	1/16	1/16	3/32	0-1t
....	0.072	...	...	3/32^l	1/8	5/32^l	3/32^l	1/8	5/32^l	...
A38	0.081	0.081	36 900	3/32	1/8	5/32	3/32	1/8	5/32	...
A47	0.091	0.093	36 600	1/8	1/8	5/32	1/8	1/8	5/32	...
A26	0.102	0.103	36 200	1/8	1/8	5/32^l	1/8	1/8	5/32	...
A52	0.128	0.128	36 900	5/32	5/32	5/32	5/32	5/32	5/32	½-1½t

Continued on p. 1060

TABLE II.—*Continued*

Designation	Nominal Thickness, in.	Actual Thickness, in.	Tensile Strength, psi.	Minimal Suitable Radius, in.						Required Bending Radii as Listed in 1944 Alcoa Handbook[a]
				For Brake Forming			For Punch Press Forming			Grain Direction Not Specified
				Bend Perpendicular to Direction of Rolling	Bend at 45 deg. to Direction of Rolling	Bend Parallel to Direction of Rolling	Bend Perpendicular to Direction of Rolling	Bend at 45 deg. to Direction of Rolling	Bend Parallel to Direction of Rolling	
ALUMINUM ALLOY 61S – T.										
Equivalent Specifications.—Federal Specification QQ-A-327, Navy Department Specification 47A12, S.A.E. Specification 281, Type 2.										
....	All thicknesses	...	42 000 min. (req'd.)	...	...	...	...	...	...	...
....	0.012	...	...	...	...	...	...	...	...	...
....	0.014	...	...	...	...	...	...	...	...	...
....	0.016	...	...	...	...	...	...	...	...	0-1t
....	0.020	...	...	$\frac{1}{32}$[c]	$\frac{1}{32}$[c]	$\frac{1}{32}$[c]	$\frac{1}{32}$[c]	$\frac{1}{32}$[c]	$\frac{1}{32}$[c]	...
....	0.025	...		$\frac{1}{32}$[c]	$\frac{1}{32}$[c]	$\frac{1}{32}$[c]	$\frac{1}{32}$[c]	$\frac{1}{32}$[c]	$\frac{1}{32}$[c]	
A63	0.032	0.032	45 400	$\frac{3}{64}$	$\frac{3}{64}$	$\frac{3}{64}$	$\frac{3}{64}$	$\frac{3}{64}$	$\frac{3}{64}$	½t-1½t
A66	0.040	0.041	43 800	$\frac{3}{64}$	$\frac{3}{64}$	$\frac{3}{64}$	$\frac{3}{64}$	$\frac{3}{64}$	$\frac{3}{64}$	...
A65	0.051	0.051	46 100	$\frac{1}{16}$	$\frac{1}{16}$	$\frac{1}{16}$	$\frac{1}{16}$	$\frac{1}{16}$	$\frac{1}{16}$	...
A64	0.064	0.063	46 000	$\frac{3}{32}$	$\frac{3}{32}$	$\frac{3}{32}$	$\frac{3}{32}$	$\frac{3}{32}$	$\frac{3}{32}$	1t-2t
A68	0.072	0.071	46 300	$\frac{3}{32}$	$\frac{3}{32}$	$\frac{3}{32}$	$\frac{3}{32}$	$\frac{3}{32}$	$\frac{3}{32}$	...
....	0.081	...	...	$\frac{1}{8}$[c]	$\frac{1}{8}$[c]	$\frac{1}{8}$[c]	$\frac{1}{8}$[c]	$\frac{1}{8}$[c]	$\frac{1}{8}$[c]	...
....	0.091	0.090	45 800	$\frac{5}{32}$	$\frac{5}{32}$	$\frac{5}{32}$	$\frac{5}{32}$	$\frac{5}{32}$	$\frac{5}{32}$	...
....	0.102	...	...	$\frac{3}{16}$[c]	$\frac{3}{16}$[c]	$\frac{3}{16}$[c]	$\frac{3}{16}$[c]	$\frac{3}{16}$[c]	$\frac{3}{16}$[c]	...
A71	0.128	0.125	44 600	$\frac{1}{4}$	$\frac{1}{4}$	$\frac{1}{4}$	$\frac{7}{32}$	$\frac{7}{32}$	$\frac{7}{32}$	1½t-3t

[a] Aluminum Company of America Handbook gives bending radii in terms of approximate sheet thickness.
[b] Actual radius was found to be sharp.
[c] Estimated.
[d] Actual radius was found to be $\frac{1}{64}$ in.
[e] 2,000 psi. above specified maximun tensile strength.
[f] Actual radius was found to be $\frac{1}{32}$ in.
[g] Actual radius was found to be $\frac{3}{32}$ in.
[h] Actual radius was found to be $\frac{7}{32}$ in.
[i] 400 psi. above specified minimum tensile limit for 52S-½H.
[j] 100 psi. above specified minimum tensile limit for 52S-½H.
[k] 400 psi. below specified minimum tensile limit for 52S-½H.
[l] Actual radius was found to be ⅛ in.

specimens occurred at $\frac{1}{2}$ to $\frac{3}{4}$ of the full load.

The specified chemical compositions of the various materials studied are given in Table I. Detailed analyses of the alloys were not made. However, spectrographic analysis, by means of a spark-comparison method, was used to confirm the identity of each lot of aluminum as to type. In a few cases this was supplemented by conventional wet methods of analysis.

TEST DATA

The tension data for the various materials studied, as well as the minimum satisfactory radii for forming 90-deg. bends in punch and die setups and on bending brakes, are given in Table II. For comparison, the table includes the radii required for forming 90-deg. bends in these materials as published in the 1944 Handbook of the Aluminum Company of America. These latter values are, however, expressed in general terms relative to the approximate thickness of the stock and usually are given as a range of values rather than a definite minimum forming radius.

The data shown in Table II represent minimum satisfactory forming radii which may be used for design. They have been corrected for inconsistencies due to observational errors in determining the end point, so that the minimum radii listed for forming successively thicker sheets of a given alloy and temper are equal to or greater than those shown for thinner material.

A study of the data shows that, with

one exception, the minimum radii required to form these aluminum alloys at a 45-deg. angle to the direction of rolling were equal to or larger than those required to form the material at a 90-deg. angle to the direction of rolling. Likewise, with one exception, the radii required to form the material parallel to the direction of rolling were equal to or larger than those required to form the material at a 45-deg. angle. Since it is a well-established fact that the best forming characteristics, particularly in cold-rolled sheet and strip, are obtained when the material is bent at right angles to the direction of rolling, these discrepancies have been corrected in Table II as indicated in the footnotes.

It will be seen from a study of the forming data that, in practically every case, the radii for punch-press work are either equal to or smaller than those shown for brake bending. This is due to the nature of the bending operations. In punch-press work the metal is drawn evenly into the die from both sides and the operation is essentially one involving bending, with little or no reduction in the thickness of the material. In brake forming, the material first starts to bend around the mandrel radius. As friction develops between the movable platen and the specimen, the latter is gripped tightly not only at the clamped end between the mandrel and the fixed platen but also at the free end between the mandrel and the movable platen. As bending progresses the metal begins to draw or "neck down" so that local reduction in thickness occurs. This action results in the need for a somewhat larger radius in brake forming than in punch-press work. However, a comparison of the two sets of data will show that this difference in all cases was small and in no case exceeded the difference in radii between two successive punches. Therefore, the minimum radii listed as suitable for brake forming also can be used for punch-press forming operations on the various sheet aluminum alloys covered by this report with little or no loss of space.

CONCLUSIONS

The studies covered by this paper indicate that:

1. The brake-forming characteristics of aluminum-alloy sheet can be evaluated by modifying a commercially available brake to provide mandrels of precise radii and a means of readily compensating for variations in sheet thickness.
2. The brake-forming characteristics of aluminum-alloy sheet compare favorably with the punch-and-die forming characteristics. Where differences exist, they are no greater than the difference between two successive radii, the larger radius being required for brake forming.

Acknowledgments:

The authors wish to thank the Bell Telephone Laboratories for permission to publish these data. We are indebted to George Scott of our Development Shops, for the original design upon which the authors' modification of the bending brake is based and to W. C. Ellis, of our Chemical Laboratories, for his helpful suggestions in the preparation of the manuscript.

DISCUSSION

Mr. E. S. Howarth[1] (*presented in written form*).—Definition of the end point in bending tests such as those reported in this paper is extremely difficult and makes comparison of work by different investigators somewhat uncertain. Permissible minimum bend radii, based on the absence of cracks in the formed specimen, depend upon whether the specimens are examined by the unaided eye, a low-magnification hand glass, or the microscope. The authors have chosen to define the end point by samples which have developed a degree of "orange peel" similar to a previously chosen standard sample. Figure 3 of the paper indicates that this condition is met some three steps larger in forming tool radius than was satisfactory to form specimens which were crack-free when examined by the unaided eye. I believe that this factor largely explains why the minimum radii for their tests, contained in Table II, are, in many instances, larger than those reported in the last column of Table II.

The information cited from the Aluminum Company of America publications is intended to be a general guide to the selection of tools for forming aluminum alloys and is based largely on the fabrication of crack-free formed articles. These publications do not show the direction of the bend with respect to the grain in the sheet material, but the radii are based on the most severe condition, namely, that in which the axis of the bend is parallel to the rolling direction.

One of the very important forming properties of sheet materials is the springback which occurs after the forming tools are separated from the work. This property is not discussed in the paper but may have been obtained from the punch-press tests in which 90-deg. punches and die were employed. If this information is available I believe it would be a very much worth-while additional contribution.

Mr. G. R. Gohn (*author's closure*).—In reply to Mr. Howarth, I can only offer our own experience on the use of forming data such as those presented in our paper. Back in 1930 the Western Electric Co. (the manufacturing unit of the Bell System) was having considerable difficulty in the manufacture of formed parts made on punch presses. Hundreds of millions of punched metal parts were made every year, but because of the lack of forming data, it was frequently necessary, after the completion of the tools, to increase tool radii to form the parts properly or to decrease the temper of the material from which the part was punched. The latter was undesirable from an engineering standpoint, the former was expensive. To reduce the amount of such work, the Western Electric Co. undertook a limited forming study of copper-base alloys.[2] That study was extended by us a few years later[3] when we studied some 20,000 different specimens using the same end point determination which is described in this paper. We repeated the tests on some of the same materials which the Western Electric Co. had studied. Despite the lack of a scientific end point we came up with almost identical minimum safe forming radii.

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[1] Chief, Metal Working Div., Aluminum Research Laboratories, New Kensington, Pa.

[2] W. A. Straw, M. D. Helfrick, and C. R. Fischrupp, "Forming Properties of Thin Sheets of Some Non-Ferrous Metals," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Inst. Metals Div., p. 317 (1931).

[3] G. R. Gohn, "The Forming Properties of Some Non-Ferrous Sheet Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 207 (1936).

Since then we have prescribed these radii for design purposes and we have had no complaints as to inability to form the parts satisfactorily. I will grant that there may be a margin of safety in our recommendations, but this has imposed no hardship on our designs. Consequently, when we undertook forming studies on aluminum alloy sheet, we used the same end point requirement as we had previously used for the copper-base alloys. We do not have any extended experience with the forming of aluminum alloys in sheet form, but we believe, in view of our experience with other non-ferrous materials, that the recommended minimum forming radii given in this paper can be safely used in design.

With respect to the question of springback in the bending brake tests, you can overform the test specimens so that the final product has a 90-deg. bend or included angle. In the punch-press test, the clamp-down pressure or the adjustment of the clearance between the punch and the die at the end of the stroke controls the amount of springback. We justify this procedure because when we make parts on a punch press a heeled tool is generally used which strikes the inside of the radius and sets the bend so that it will have the prescribed angle.

ATMOSPHERIC AND INDOOR AGING STUDIES ON SOME ALUMINUM AND ZINC-BASE DIE-CASTING ALLOYS*

BY G. R. GOHN [1] AND LUCILLE E. MENGES [1]

SYNOPSIS

The results of indoor and outdoor aging studies covering a period of 15 yr. on twelve aluminum and nine zinc-base die-casting alloys are presented in this paper. These alloys were die-cast by a number of producers for a cooperative investigation of various die-casting alloys originally undertaken by the former Subcommittee XV on Die-Cast Metals and Alloys, of A.S.T.M. Committee B-2 and carried to completion by the subcommittee's successor, Committee B-6 on Die-Cast Metals and Alloys.

At the time these studies were undertaken in 1927, the various specimens represented the best commercial practices with respect to composition and methods of die-casting; but in the intervening years, technological improvements in the alloys and equipment (many of them the outgrowth of these cooperative studies) as well as changes in commercial die-casting practices have made obsolete all of the zinc-base die-casting alloys and all but three of the aluminum-base die-casting alloys. Only aluminum-base alloys Nos. IV, V and a modification of VII are in commercial use today.

The studies indicate that there was very little difference in the effect of indoor or outdoor aging on the physical properties of the twelve aluminum-base alloys during the 15-yr. test period. On the other hand, all of the nine zinc-base alloys showed marked losses in physical properties during the same period of time. For most of the zinc-base alloys, the losses on outdoor aging were only slightly greater than those shown in the indoor aging tests. In the case of zinc-base alloys having a nominal composition of 4 per cent aluminum, 3 per cent copper, remainder zinc, the losses in physical properties were reduced when a higher quality of zinc was used as the basis metal. Small additions of magnesium also retarded the losses. The greatest resistance to aging was shown by the zinc-base alloy containing 4 per cent aluminum, 3 per cent copper, compounded from Special High-Grade Zinc (minimum, 99.98 per cent zinc) with added magnesium.

In 1927 when Subcommittee XV (now Committee B-6) of A.S.T.M. Committee B-2 on Non-Ferrous Metals and Alloys undertook a comprehensive series of cooperative tests on both aluminum and zinc-base die-casting alloys, the Bell Telephone Laboratories participated in the program as one of the testing laboratories. Space was provided on the roof of their New York City building for the outdoor exposure racks of the committee. In addition to their participation in the work of the committee, the Bell Telephone Laboratories carried out a series of parallel indoor and outdoor aging studies on all of the alloys die-cast by the various producers. The latter tests, which were made on specimens supplied by the subcommittee, covered exposure periods of 6 months to 15 yr. and reflect the effect of an accelerated outdoor exposure not used in the committee's tests. These studies have now been completed and, although all of the zinc-base alloys and all but three of the aluminum-base alloys are now obsolete, the data are presented in this paper to supplement those appearing in the annual reports of the committee.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

[1] Bell Telephone Laboratories, Inc., New York, N. Y.

TESTING PROGRAM

The specimens used in these studies were die-cast from twelve aluminum-base alloys by five different producers and from nine zinc-base alloys by two to six different producers, depending upon the alloy. Details concerning the original choice of alloys, a description of the basis metals used in preparing the alloys and a list of the producers die-casting test specimens are given in the 1928 Report of Subcommittee XV, of A.S.T.M. Committee B-2 (**1**).[2]

The testing program consisted of tension tests on $\frac{1}{4}$-in. diameter round tension specimens in the "as cast" condition, and after shelf-life or indoor aging for periods of 6 months, 1, 2, 5, and 15 yr.; tension tests on $\frac{1}{8}$ by $\frac{1}{2}$-in. flat tension specimens in the "as cast" condition, and after 6 months, 2, 5, and 15 yr. of outdoor exposure; and Charpy impact tests on $\frac{1}{4}$ by $\frac{1}{4}$-in. square, unnotched test bars after 15 yr. of shelf-life aging. All exposure tests, both indoors and outdoors, were made in New York City. Because of a shortage of test specimens, the zinc-base alloys were not tested after the 5-yr. indoor or outdoor exposure period.

METHODS OF TEST

The methods of test used in these studies were essentially those outlined in Appendix III to the 1928 Report of Subcommittee XV (**2**). The tension specimens were tested at a maximum cross-head speed of 0.025 in. per inch of free length, on a 20,000-lb. Amsler universal testing machine. The scale used was selected to give fracture at one half to three quarters of the full-scale reading. This machine was accurate to ±1 per

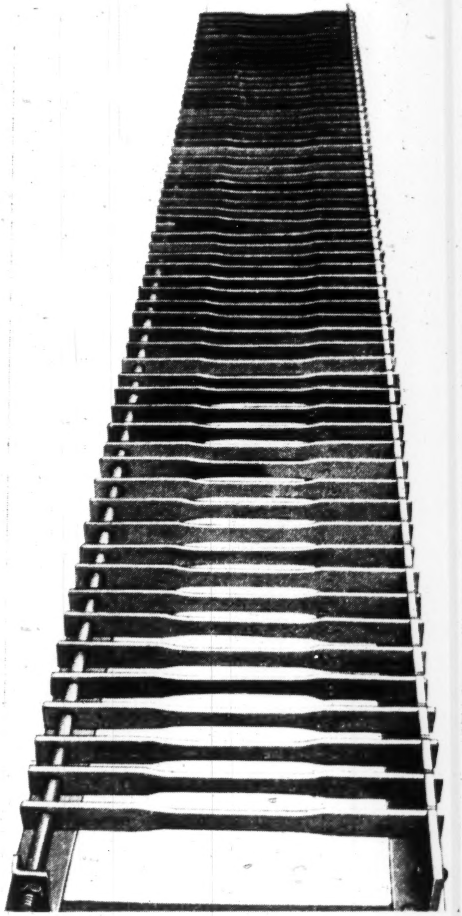

FIG. 1.—Die-Cast Specimens Mounted on Test Rack.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1094.

cent at any load. The percentage elongation was determined with the use of dividers to the nearest 0.5 per cent. In most cases ten specimens were used for each test.

The Charpy impact values were determined on a 30 ft-lb. Riehle impact tester. The ten 6-in. test bars were cut in the center and impact tests were made on both the vent and gate ends, using unnotched specimens.

The die-cast test specimens were mounted on racks as shown in Fig. 1, and the individual racks were mounted on frames as shown in Fig. 2 (see also Fig. 6 of the 1930 Report of Subcommittee XV of Committee B-2 (3)). Whereas the specimens exposed for Subcommittee XV were mounted in a manner similar to that shown in Fig. 1 and exposed to normal atmospheric corrosion at an elevation of 150 ft., the specimens used in these studies were exposed to an "artificial rain" at an elevation of 50 ft.

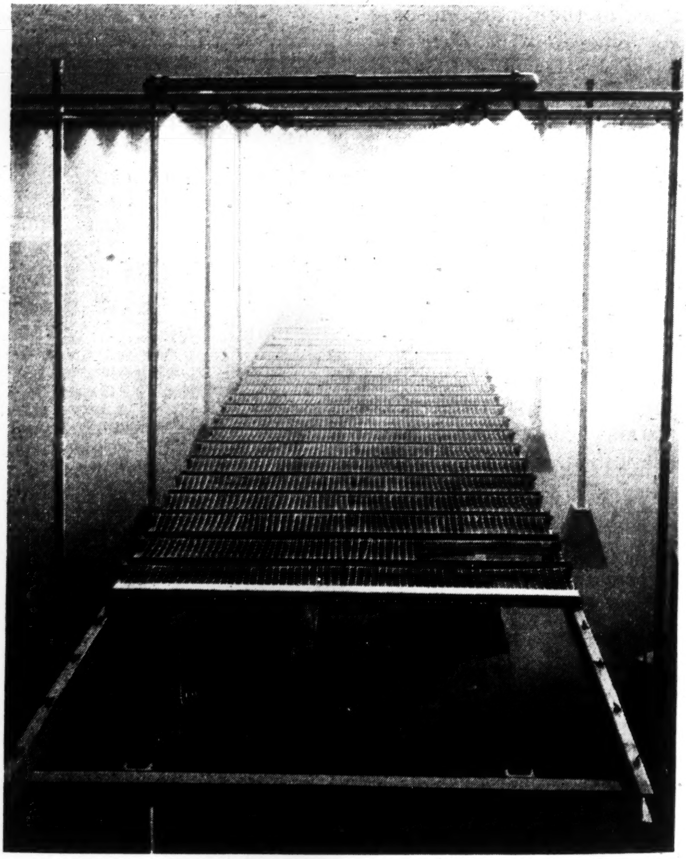

FIG. 2.—Test Racks Exposed to "Artificial Rain."

In both cases the exposure racks were located on the roof of the Bell Telephone Laboratories building at 463 West St., directly opposite large steamship docks on the Hudson River. Consequently, in addition to the smoke and dust normally encountered in a large, industrial, seacoast city, the atmosphere in the immediate vicinity of the exposure site receives considerable additional smoke contamination from steamships tied up at the docks and from tugs and other vessels in the heavy river traffic passing this point. The sulfur dioxide content of the air is also considerably above that normally found in city atmospheres and a relatively high percentage of dust is present. This dust consists largely of carbonaceous matter, primarily cinders, together with smaller percentages of soot and coal dust. According to Hippensteel, Borgmann and Farnsworth (**4**) these particles add to the corrosiveness of the environment in that they may form nuclei for local attack. The atmosphere to which the test specimens were exposed may therefore be considered as highly industrial in character and severely corrosive.

The severity of the exposure was increased by spraying the specimens with water. This spray or "artificial rain" was intended to simulate additional rainfall, with the expectation of obtaining a reasonable acceleration of the normal weathering. It was applied three times daily (except Sunday) for 15-min. periods during the 8 months from April to December of each year until 1937 when the "artificial rain" system was discontinued because the acceleration was not as great as anticipated. The aluminum-base alloys were exposed to the accelerated corrosion just described for the first 9 yr. of the test period, the zinc-base alloys for the first 6 yr. This difference was due to the fact that the zinc-base alloys were not available when the tests were started on the aluminum-base alloys. Other climatic details applicable to the test site are given in Table XXII taken from the 1932 Report of Subcommittee VI of A.S.T.M. Committee B-3 (**5**).

TEST DATA

Tables I to XXI, inclusive, list the nominal chemical compositions and the coordinated chemical analyses for each of the alloys based upon the analytical results from several laboratories, as well as the average tensile strength and percentage elongation in 2 in. after various testing periods. Because the shape of the test specimen determines, to some extent, the resulting physical properties it was necessary to determine the tensile properties of both the round and flat tension specimens in the "as cast" condition, that is at zero time, as separate reference values for comparing the effect of various indoor and outdoor aging periods. The chemical analyses are taken from the 1935 Report of Subcommittee V of A.S.T.M. Committee B-6 (**6**). Charpy impact data for specimens "as cast" and after aging outdoors for 15 yr. are also given in Tables I to XXI, inclusive. Inasmuch as the initial impact values, as determined by the Bell Telephone Laboratories, were Izod values, the "as cast" Charpy impact data given in Tables I to XXI are taken from the tables on pp. 197 to 212 of Appendix II to the 1935 Report of Subcommittee V (**6**).

Other pertinent data on these alloys including metallographic and X-ray studies (**7**, **8**, **9**), density determinations (**10**), methods of statistical analysis (**11**), discussions on the expansion test for zinc-base die-castings (**12**), the effect of composition on aluminum-base die castings (**13**), and the results of the committee studies on these alloys after 1 and 5-yr. exposure for the aluminum-base alloys (**14**, **15**), and after 1, 5, and 10-yr. exposure for the zinc-base alloys (**14**, **15**, **16**) are given in the various reports of A.S.T.M. Committees B-2 and B-6, and their subcommittees.

DISCUSSION OF DATA

Aluminum-Base Alloys:

A study of the test data given in Tables I to XII for the aluminum-base alloys (see also Figs. 3 to 14 inclusive) indicates that after 15 yr. of indoor aging, all twelve of the alloys show a slight increase in tensile strength. This increase in tensile strength varies from less than 1 per cent for the high-silicon alloy (No. V) to approximately 10 per cent, both, for the low-copper, silicon alloy (No. VI), and for alloys containing 4 per cent or more of copper, with or without other added elements such as silicon or nickel (alloys Nos. I, II, III, VII, IX, and XII). The silicon-nickel alloy (No. X) showed only slightly greater change in tensile strength than did the high-silicon alloy (No. V). The low-silicon alloy (No. IV), the low-copper, silicon-nickel alloy (No. VIII), and the low-copper, high-silicon alloy (No. XI) all showed similar aging characteristics, the increase in tensile strength being about 6 per cent.

During the same period of time the elongation of these alloys was also affected, the changes varying from an increase of approximately 15 per cent for the high-silicon alloy (No. V), the low-copper, silicon-nickel alloy (No. IX), and the silicon-nickel alloy (No. X) to a decrease of about 25 per cent for the three alloys containing 8 per cent or more of copper (alloys Nos. II, III, and XII). The impact strength of all of the alloys, except the high-silicon alloy (No. V), decreased by amounts varying from 14 to 48 per cent. The latter figure corresponds to a loss of 1.2 ft-lb. In the case of the high-silicon alloy (No. V) there was no change in impact strength after 15 yr. of indoor aging.

The data show that, in the indoor aging tests, there was very little change in tensile strength after the 2-yr. exposure period. There was, however, a continuing loss in elongation after the 2-yr. test period.

A study of the test data given in Tables I to XII (see also Figs. 3 to 14 inclusive) indicates that, after 15 yr. of outdoor aging, the only aluminum-base alloys which showed no increase in tensile strength were the 4 per cent copper alloy (No. I), the low and the high-silicon alloys (Nos. IV and V), and the silicon-nickel alloy (No. X). For these alloys, losses in tensile strength of 1 to 8 per cent were observed. The greatest loss in tensile strength occurred in the case of the high-silicon alloy (No. V). The 10 per cent copper alloy (No. II), the low-copper, silicon alloy (No. VI), the low-copper, high-silicon alloy (No. XI), the high-copper, low-silicon (No. XII), and the low-copper, silicon-nickel alloy (No. VIII) all showed increases in tensile strength of 1 per cent or less. The remaining alloys, including the 14 per cent copper alloy (No. III), the 4 per cent copper, 5 per cent silicon alloy (No. VII), and the low-copper, silicon-nickel alloy (No. IX), showed increases in tensile strength of 2 to 4 per cent.

The changes in elongation during the 15 yr. of outdoor exposure varied from an increase of 6 per cent for the low-copper, high-silicon alloy (No. XI) to a loss of 33 per cent for the 14 per cent copper alloy (No. III). There was no change in elongation for the high-silicon alloy (No. V) or for the low-copper, silicon-nickel alloy (No. IX). Alloys containing only copper or copper with small additions of silicon (Nos. I, II, III, and XII) showed the greatest loss in elongation although the changes were not substantially greater than those observed for the other alloys such as Nos. VI, VII, and VIII containing smaller amounts of copper.

As in the case of the indoor aging studies, the bulk of the changes in ten-

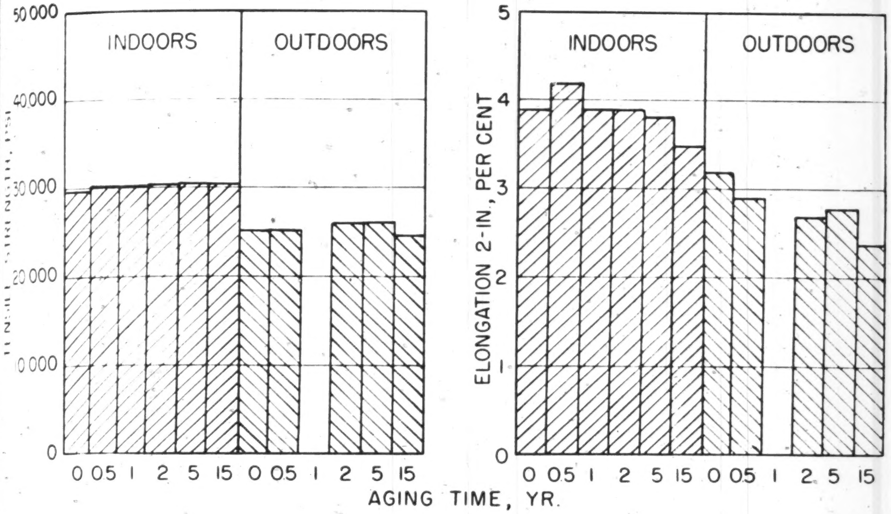

FIG. 3.—Aging Characteristics of Aluminum-Base Alloy No. I.

TABLE I.—ALUMINUM-BASE ALLOY NO. I.

Producer	Coordinated Chemical Analyses—All Laboratories, per cent								
	Copper	Silicon	Nickel	Iron	Manganese	Magnesium	Zinc	Aluminum	Other Elements
Nominal Composition	4.0	....	....	1.5 to 2.0	0.05 Max.	0.05 Max.	....	Remainder	....
C	4.37	0.43	nil	1.48	0.01	0.03	0.05	93.63	nil
D	3.51	0.63	0.17	1.50	0.03	0.03	0.18	93.95	nil
G	3.97	0.32	0.03	2.77	0.02	0.01	nil	92.80	tin 0.08
S	4.23	0.59	0.45	1.90	0.02	0.04	0.03	92.74	nil
W	3.80	0.31	nil	2.25	0.03	nil	0.08	93.43	nil

Producer	INDOOR EXPOSURE AT NEW YORK, N. Y.						OUTDOOR EXPOSURE AT NEW YORK, N. Y.				
	As Cast	6 Months	1 yr.	2 yr.	5 yr.	15 yr.	As Cast	6 Months	2 yr.	5 yr.	15 yr.
	Tensile Strength, psi. (¼-in. Diameter Round Bars)						Tensile Strength, psi. (⅛ by ½-in. Flat Bars)				
C	29 310	29 480	30 360	30 220	30 860	31 380	23 860	21 650	23 130	25 270	22 420
D	24 600	28 950	26 820	29 850	29 160	28 600	24 010	26 000	26 620	25 370	24 890
G	28 210	29 200	31 940	32 070	31 470	31 960	24 490	26 460	27 990	27 520	27 290
S	34 330	34 600	33 290	35 240	36 680	36 940	24 770	24 070	24 220	22 870	21 350
W	31 990	33 340	32 840	33 160	34 170	34 020	29 770	28 890	28 790	29 930	28 310
Average	29 690	31 110	31 050	32 110	32 470	32 580	25 380	25 410	26 150	26 190	24 850
	Elongation in 2 in., per cent (¼-in. Diameter Round Bars)						Elongation in 2 in., per cent (⅛ by ½-in. Flat Bars)				
C	4.7	4.6	4.4	4.5	3.4	3.3	3.1	2.3	2.1	3.1	2.2
D	2.8	4.2	3.3	3.9	3.4	3.2	3.7	2.7	2.7	2.8	2.2
G	2.9	3.3	3.0	3.1	3.9	2.4	3.3	3.0	2.2	2.3	1.9
S	4.4	5.3	4.5	3.8	5.3	4.7	3.0	2.3	2.2	2.0	2.0
W	4.5	3.8	4.2	4.2	3.0	3.8	3.0	4.0	4.4	3.8	3.5
Average	3.9	4.2	3.9	3.9	3.8	3.5	3.2	2.9	2.7	2.8	2.4
	Charpy Impact Value, ft-lb. (¼-in. Square Bars)										
C	...					4.2					
D	...					5.5					
G	5.4					3.7					
S	7.5					6.7					
W	6.5					...					
Average	6.5					5.0					

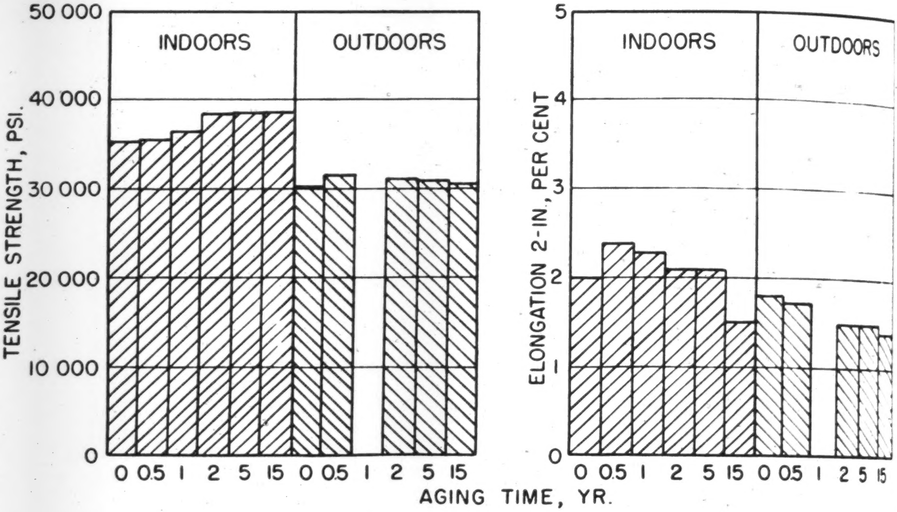

FIG. 4.—Aging Characteristics of Aluminum-Base Alloy No. II.

TABLE II.—ALUMINUM-BASE ALLOY NO. II.

Producer	Coordinated Chemical Analyses—All Laboratories, per cent								
	Copper	Silicon	Nickel	Iron	Manganese	Magnesium	Zinc	Aluminum	Other Elements
Nominal Composition	10.0	....	....	1.5 to 2.0	0.05 Max.	0.05 Max.	....	Remainder	....
C	7.13	0.38	nil	1.96	0.01	0.04	0.16	90.52	nil
D	8.97	0.34	0.05	1.44	0.03	0.13	0.09	88.95	nil
G	9.36	0.31	nil	1.71	0.02	nil	nil	88.60	nil
S	10.47	0.44	nil	1.44	0.01	0.01	0.05	87.58	nil
W	8.93	0.44	nil	3.07	0.03	nil	0.20	87.33	nil

Producer	INDOOR EXPOSURE AT NEW YORK, N. Y.						OUTDOOR EXPOSURE AT NEW YORK, N. Y.				
	As Cast	6 Months	1 yr.	2 yr.	5 yr.	15 yr.	As Cast	6 Months	2 yr.	5 yr.	15 yr.
	Tensile Strength, psi. (¼-in. Diameter Round Bars)						Tensile Strength, psi. (⅛ by ½-in. Flat Bars)				
C	34 860	33 720	36 490	37 430	38 540	38 850	31 970	30 600	30 130	28 370	28 460
D	34 760	34 490	36 200	39 060	38 860	35 320	29 080	32 220	32 650	30 920	30 140
G	34 600	34 920	34 800	36 970	37 310	38 680	29 290	29 640	30 940	29 810	30 870
S	34 750	37 220	36 870	37 710	38 460	39 620	25 080	29 170	25 800	30 660	28 540
W	36 640	37 220	37 750	40 290	38 910	40 300	35 420	36 540	35 650	35 190	34 660
Average	35 120	35 510	36 420	38 290	38 420	38 550	30 170	31 630	31 030	30 990	30 530
	Elongation in 2 in., per cent (¼-in. Diameter Round Bars)						Elongation in 2 in., per cent (⅛ by ½-in. Flat Bars)				
C	3.2	3.8	2.7	3.1	2.0	2.2	2.4	3.1	2.0	2.0	1.9
D	1.0	1.0	1.4	2.0	2.0	1.5	1.0	0.8	1.2	2.0	0.9
G	2.9	3.2	2.9	2.0	3.0	1.5	2.8	2.2	1.6	1.3	1.5
S	1.0	2.0	2.6	1.4	1.5	1.5	1.1	1.1	1.1	1.0	1.0
W	2.0	2.0	1.8	1.9	1.9	1.0	1.5	1.5	1.4	1.0	1.8
Average	2.0	2.4	2.3	2.1	2.1	1.5	1.8	1.7	1.5	1.5	1.4
	Charpy Impact Value, ft-lb. (¼-in. Square Bars)										
C	4.0					2.6					
D	[a]					1.9					
G	4.1					2.7					
S	3.4					2.3					
W	2.6					1.7					
Average	3.5					2.2					

[a] Test data not available.

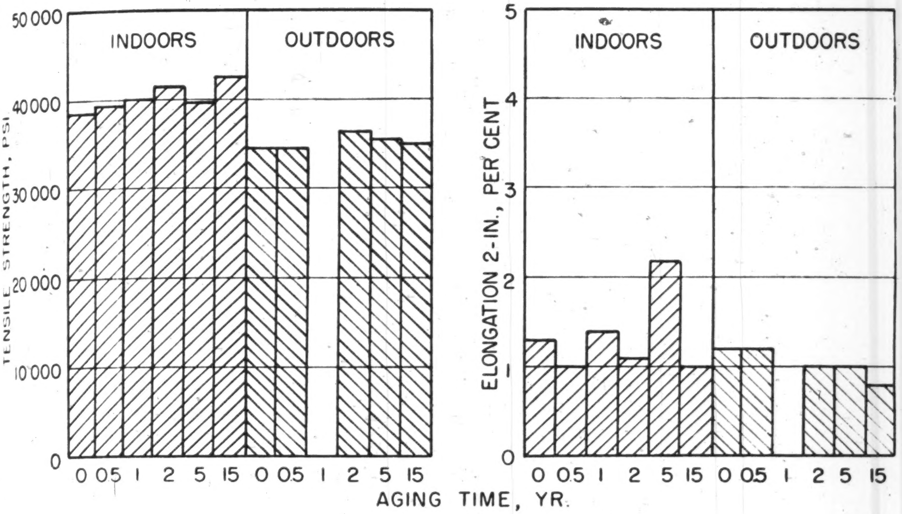

FIG. 5.—Aging Characteristics of Aluminum-Base Alloy No. III.

TABLE III.—ALUMINUM-BASE ALLOY NO. III.

Producer	Coordinated Chemical Analyses—All Laboratories, per cent								
	Copper	Silicon	Nickel	Iron	Manganese	Magnesium	Zinc	Aluminum	Other Elements
Nominal Composition...	14.0	....	....	1.5 to 2.0	0.05 Max.	0.05 Max.	....	Remainder	....
C..........	11.99	0.43	0.14	1.76	0.02	0.03	0.09	85.54	nil
D..........	13.14	0.32	0.12	1.67	0.08	0.03	0.09	84.55	nil
G..........	14.29	0.27	nil	1.72	0.02	0.01	0.03	83.68	nil
S..........	13.20	0.53	nil	1.57	0.01	0.01	0.08	84.60	nil
W..........	13.02	0.37	nil	2.74	0.03	0.02	0.11	83.71	nil

Producer	INDOOR EXPOSURE AT NEW YORK, N. Y.						OUTDOOR EXPOSURE AT NEW YORK, N. Y.				
	As Cast	6 Months	1 yr.	2 yr.	5 yr.	15 yr.	As Cast	6 Months	2 yr.	5 yr.	15 yr.
	Tensile Strength, psi. (¼-in. Diameter Round Bars)						Tensile Strength, psi. (⅛ by ½-in. Flat Bars)				
C..........	36 820	38 200	40 160	42 190	33 950	43 740	33 630	35 580	36 420	36 380	34 350
D..........	38 020	38 920	40 480	40 750	41 050	39 080	35 180	33 300	36 800	31 380	34 320
G..........	34 470	38 580	38 030	41 890	39 380	40 620	32 800	34 500	36 730	37 570	36 200
S..........	40 580	39 640	40 150	39 110	42 020	43 900	32 820	33 590	34 590	32 660	33 810
W..........	41 580	40 700	41 350	41 900	41 810	43 410	37 030	34 500	36 510	38 070	36 130
Average..........	38 290	39 210	40 030	41 170	39 640	42 150	34 290	34 290	36 210	35 210	34 960
	Elongation in 2 in., per cent (¼-in. Diameter Round Bars)						Elongation in 2 in., per cent (⅛ by ½-in. Flat Bars)				
C..........	1.6	1.5	1.4	2.0	6.4	1.0	1.0	2.2	1.0	1.3	1.3
D..........	0.5	0.5	1.0	0.6	0.9	0.5	0.5	0.8	1.0	1.0	0.6
G..........	3.0	0.9	1.9	0.9	1.2	1.8	3.0	1.0	1.2	0.7	0.5
S..........	1.0	1.1	1.5	0.6	1.4	1.0	1.0	1.1	1.0	1.0	1.0
W..........	0.5	1.0	1.4	1.4	1.0	0.7	0.6	1.1	1.0	1.0	0.6
Average	1.3	1.0	1.4	1.1	2.2	1.0	1.2	1.2	1.0	1.0	0.8
	Charpy Impact Value, ft-lb. (¼-in. Square Bars)										
C..........	[a]					1.2					
D..........	[a]					1.0					
G..........	2.2					0.8					
S..........	2.4					1.7					
W..........	1.8					[b]					
Average..........	2.1					1.2					

[a] Test data not available.

[b] No test specimens available.

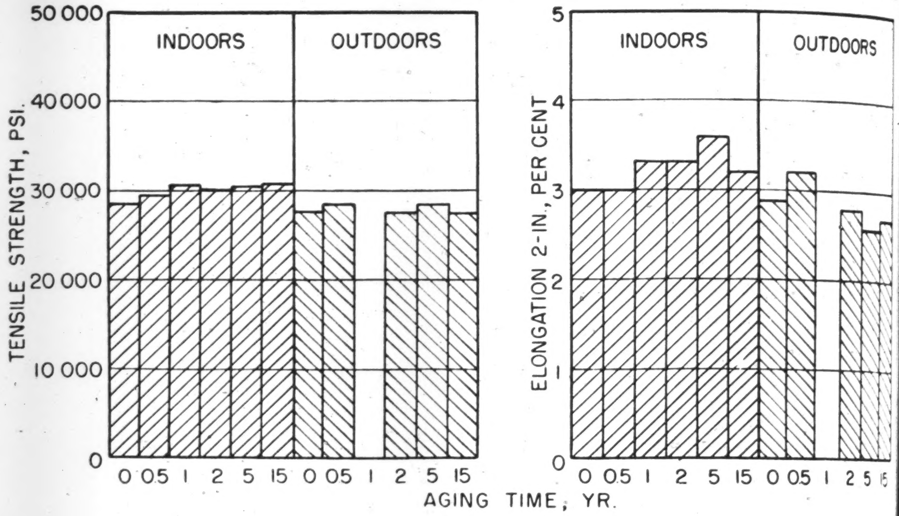

Fig. 6.—Aging Characteristics of Aluminum-Base Alloy No. IV.

TABLE IV.—ALUMINUM-BASE ALLOY NO. IV.

Producer	Coordinated Chemical Analyses—All Laboratories, per cent								
	Copper	Silicon	Nickel	Iron	Manganese	Magnesium	Zinc	Aluminum	Other Elements
Nominal Composition	....	5.0	....	1.5 to 2.0	0.05 Max.	0.05 Max.	....	Remainder	...
C	0.58	4.16	0.01	1.85	0.01	0.07	0.02	93.30	nil
D	0.34	5.07	nil	1.48	0.07	0.06	0.44	92.54	nil
G	0.68	5.27	nil	1.29	0.02	0.01	nil	92.73	nil
S	0.24	5.11	0.22	1.89	0.04	0.01	0.11	92.38	nil
W	0.61	4.41	nil	2.53	0.05	0.02	5.90	86.50	nil

Producer	Indoor Exposure at New York, N. Y.						Outdoor Exposure at New York, N. Y.				
	As Cast	6 Months	1 yr.	2 yr.	5 yr.	15 yr.	As Cast	6 Months	2 yr.	5 yr.	15 yr.
	Tensile Strength, psi. (¼-in. Diameter Round Bars)						Tensile Strength, psi. (⅛ by ½-in. Flat Bars)				
C	30 120	30 920	31 230	31 350	32 170	31 400	29 480	29 800	29 250	28 260	28 690
D	25 680	26 690	26 400	25 580	26 800	26 660	23 220	25 310	23 890	26 240	23 360
G	29 250	30 440	31 350	31 130	31 140	30 460	28 170	27 670	27 840	29 040	27 310
S	28 900	31 720	31 250	29 980	31 010	31 820	28 690	29 460	26 920	29 610	27 990
W	30 500	29 120	33 090	32 200	31 700	33 340	29 440	31 080	30 220	29 930	30 310
Average	28 890	29 780	30 660	30 050	30 560	30 740	27 800	28 660	27 620	28 620	27 540
	Elongation in 2 in., per cent (¼-in. Diameter Round Bars)						Elongation in 2 in., per cent (⅛ by ½-in. Flat Bars)				
C	4.5	4.3	4.4	4.4	5.1	3.8	3.7	3.9	3.7	3.0	3.3
D	2.3	2.6	2.9	2.3	2.6	2.8	2.4	2.8	2.0	1.5	2.0
G	3.0	3.1	4.2	4.2	4.2	4.0	3.0	3.9	3.4	3.9	3.4
S	3.1	3.9	3.3	4.0	3.6	3.7	3.0	3.4	2.8	2.8	3.0
W	2.0	1.3	1.9	1.5	2.6	1.7	2.6	2.0	2.2	1.8	2.0
Average	3.0	3.0	3.3	3.3	3.6	3.2	2.9	3.2	2.8	2.6	2.7
	Charpy Impact Value, ft-lb. (¼-in. Square Bars)										
C	4.3					3.4					
D	[a]					3.0					
G	5.7					4.2					
S	6.3					4.5					
W	2.6					1.8					
Average	**4.7**					**3.4**					

[a] **Test data not available.**

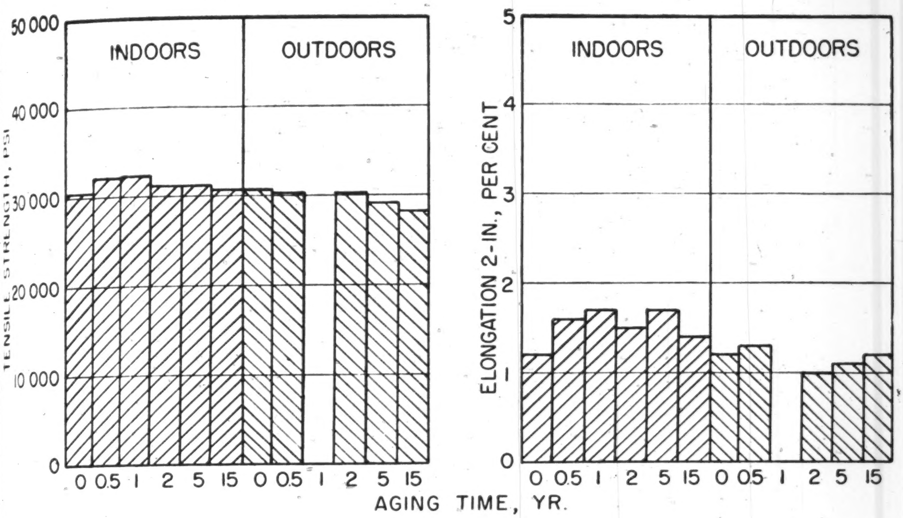

FIG. 7.—Aging Characteristics of Aluminum-Base Alloy No. V.

TABLE V.—ALUMINUM-BASE ALLOY NO. V.

Producer	Coordinated Chemical Analyses—All Laboratories, per cent								
	Copper	Silicon	Nickel	Iron	Manganese	Magnesium	Zinc	Aluminum	Other Elements
Nominal Composition...	....	13.0	....	1.5 to 2.0	0.05 Max.	0.05 Max.	....	Remainder	....
C..........	0.64	13.0	0.08	1.59	0.19	0.09	0.22	84.20	nil
D..........	0.98	12.96	0.03	1.52	0.09	0.12	0.90	83.40	nil
G..........	0.15	12.42	nil	1.89	0.03	0.01	0.03	85.20	tin 0.06, lead 0.21
S..........	0.05	12.97	0.02	1.35	0.02	0.03	0.06	85.50	nil
W..........	0.20	10.72	nil	4.34	0.06	0.04	0.60	84.04	nil

Producer	INDOOR EXPOSURE AT NEW YORK, N. Y.						OUTDOOR EXPOSURE AT NEW YORK, N. Y.				
	As Cast	6 Months	1 yr.	2 yr.	5 yr.	15 yr.	As Cast	6 Months	2 yr.	5 yr.	15 yr.
	Tensile Strength, psi. (¼-in. Diameter Round Bars)						Tensile Strength, psi. (⅛ by ½-in. Flat Bars)				
C..........	34 020	35 180	35 610	35 570	33 530	34 320	34 140	31 600	33 800	32 270	30 850
D..........	25 810	27 220	29 500	28 090	28 160	24 330	*a*	28 400	27 500	25 570	25 600
G..........	28 710	30 800	31 520	29 840	29 330	28 790	28 320	28 380	30 060	27 400	27 040
S..........	34 650	37 850	37 440	36 060	35 730	36 720	31 970	34 950	31 200	33 200	28 740
W..........	29 670	29 760	27 230	27 700	30 500	29 500	28 780	28 910	29 360	27 280	28 900
Average..........	30 570	32 160	32 260	31 450	31 450	30 730	30 800	30 450	30 380	29 140	28 230
	Elongation in 2 in., per cent (¼-in. Diameter Round Bars)						Elongation in 2 in., per cent (⅛ by ½-in. Flat Bars)				
C..........	1.3	1.6	1.5	1.2	1.1	1.3	1.5	1.7	0.5	1.0	0.9
D..........	0.9	0.5	1.5	1.2	1.7	1.1	1.0	1.3	1.0	1.0	1.5
G..........	1.0	2.0	2.0	1.5	2.0	1.5	0.9	1.1	1.0	1.0	1.2
S..........	2.5	2.2	1.9	2.3	2.1	2.1	1.4	1.9	1.6	1.8	1.7
W..........	0.5	1.5	1.5	1.1	1.4	0.8	1.0	0.7	0.8	0.9	0.9
Average..........	1.2	1.6	1.7	1.5	1.7	1.4	1.2	1.3	1.0	1.1	1.2
	Charpy Impact Value, ft-lb. (¼-in. Square Bars)										
C..........	*a*					*b*					
D..........	*a*					1.3					
G..........	1.17					*b*					
S..........	*a*					*b*					
W..........	1.5					*b*					
Average..........	1.3					1.3					

[a] Test data not available.
[b] No test specimens available.

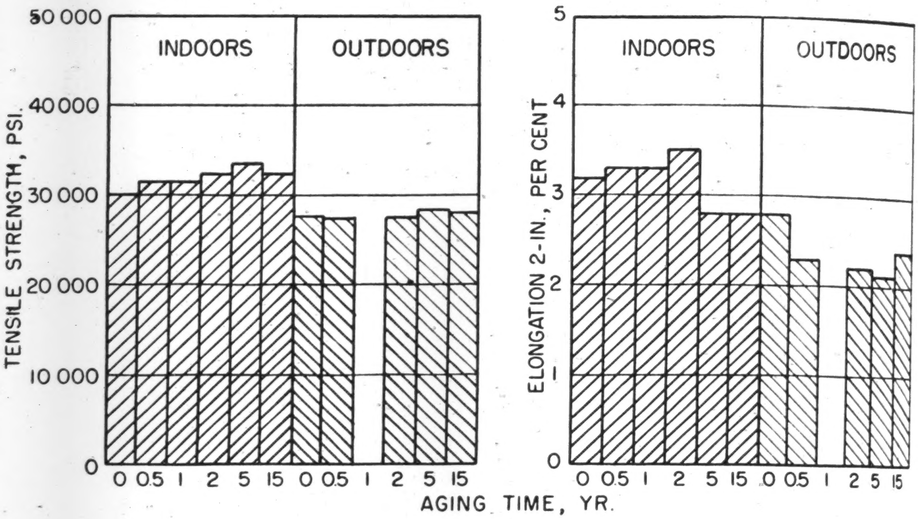

Fig. 8.—Aging Characteristics of Aluminum-Base Alloy No. VI.

TABLE VI.—ALUMINUM-BASE ALLOY NO. VI.

Producer	Copper	Silicon	Nickel	Iron	Manganese	Magnesium	Zinc	Aluminum	Other Elements
	Coordinated Chemical Analyses—All Laboratories, per cent								
Nominal Composition	2.0	3.0	....	1.5 to 2.0	0.05 Max.	0.05 Max.	....	Remainder	....
C	1.91	2.96	0.35	1.30	0.66	0.05	0.42	92.35	nil
D	1.98	3.19	nil	1.53	0.04	0.07	0.33	92.87	nil
G	2.06	3.23	nil	1.98	0.03	0.01	0.01	92.68	nil
S	2.18	3.27	nil	1.86	0.02	0.02	0.04	92.61	nil
W	2.16	5.02	nil	3.38	0.06	0.01	0.16	89.21	nil

Producer	Indoor Exposure at New York, N. Y.						Outdoor Exposure at New York, N. Y.				
	As Cast	6 Months	1 yr.	2 yr.	5 yr.	15 yr.	As Cast	6 Months	2 yr.	5 yr.	15 yr.
	Tensile Strength, psi. (¼-in. Diameter Round Bars)						Tensile Strength, psi. (⅛ by ½-in. Flat Bars)				
C	30 100	30 820	31 140	32 920	37 160	31 880	25 140	26 700	25 490	26 560	24 840
D	26 460	28 880	29 200	29 990	28 750	28 790	25 940	23 900	26 100	27 650	25 040
G	29 830	29 740	30 730	32 070	32 910	32 750	26 470	26 510	26 820	27 160	27 710
S	31 240	33 120	32 790	32 980	34 180	34 090	29 780	29 400	30 770	31 100	30 840
W	32 620	34 940	33 870	33 210	34 310	34 950	32 440	31 500	29 970	30 340	32 020
Average	30 050	31 500	31 550	32 230	33 460	32 490	27 950	27 600	27 830	28 560	28 090
	Elongation in 2 in., per cent (¼-in. Diameter Round Bars)						Elongation in 2 in., per cent (⅛ by ½-in. Flat Bars)				
C	3.7	3.0	3.7	3.8	1.4	3.2	2.3	2.6	1.3	3.5	2.1
D	3.3	3.3	2.9	3.4	2.5	1.0	2.7	2.1	2.3	2.5	2.2
G	3.2	3.7	4.0	4.5	4.2	4.3	2.9	2.8	2.5	2.0	2.7
S	4.0	4.4	4.3	4.0	3.7	3.7	4.2	2.5	3.4	1.0	3.7
W	1.9	2.1	1.8	1.6	2.2	1.7	1.7	1.7	1.5	1.5	1.4
Average	3.2	3.3	3.3	3.5	2.8	2.8	2.8	2.3	2.2	2.1	2.4
	Charpy Impact Value, ft-lb. (¼-in. Square Bars)										
C	[a]					3.5					
D	[a]					3.8					
G	6.1					3.9					
S	5.5					1.6					
W	2.4					1.7					
Average	4.7					2.9					

[a] Test data not available.

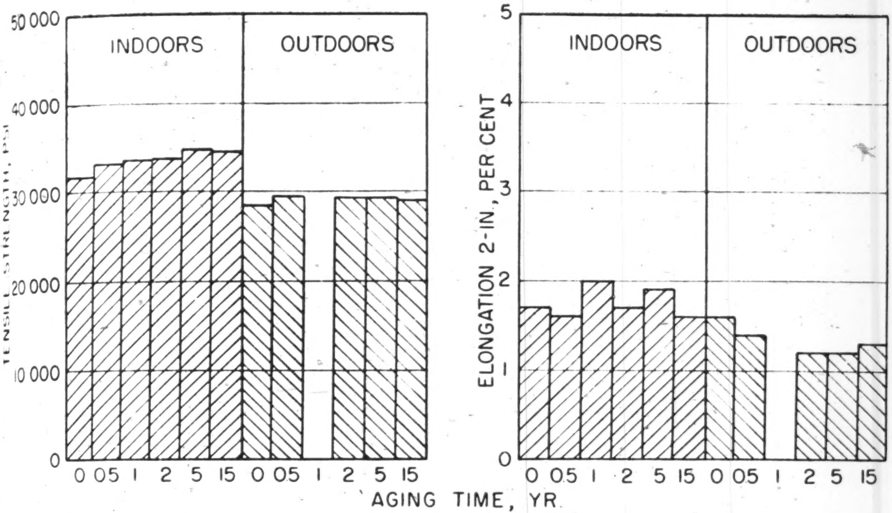

FIG. 9.—Aging Characteristics of Aluminum-Base Alloy No. VII.

TABLE VII.—ALUMINUM-BASE ALLOY NO. VII.

Producer	Coördinated Chemical Analyses—All Laboratories, per cent								
	Copper	Silicon	Nickel	Iron	Manganese	Magnesium	Zinc	Aluminum	Other Elements
Nominal Composition...	4.0	5.0	....	1.5 to 2.0	0.05 Max.	0.05 Max.	....	Remainder	....
C............	3.53	5.11	0.09	1.45	0.20	0.01	0.43	89.18	nil
D............	3.86	5.18	0.04	1.51	0.05	0.04	0.38	88.94	nil
G............	3.92	4.83	nil	1.56	0.16	nil	0.08	88.11	tin 0.76, lead 0.48
S............	3.78	5.08	0.14	1.86	0.02	0.01	0.12	89.21	nil
W............	3.53	5.70	nil	3.07	0.05	nil	0.22	87.43	nil

Producer	INDOOR EXPOSURE AT NEW YORK, N. Y.						OUTDOOR EXPOSURE AT NEW YORK, N. Y.				
	As Cast	6 Months	1 yr.	2 yr.	5 yr.	15 yr.	As Cast	6 Months	2 yr.	5 yr.	15 yr.
	Tensile Strength, psi. (¼-in. Diameter Round Bars)						Tensile Strength, psi. (⅛ by ½-in. Flat Bars)				
C............	35 400	35 980	36 220	37 460	36 800	38 800	30 280	30 140	30 630	30 800	29 070
D............	28 070	31 820	31 720	31 860	33 080	31 460	28 150	28 730	28 370	30 710	28 950
G............	29 250	29 400	31 000	30 380	32 450	32 880	27 360	26 590	27 490	25 430	26 510
S............	33 800	35 340	36 100	35 230	38 320	36 310	26 340	31 580	30 050	28 590	30 440
W............	32 820	32 860	32 870	33 760	34 110	33 680	30 390	30 880	29 490	30 610	30 410
Average............	31 870	33 080	33 580	33 740	34 950	34 630	28 500	29 580	29 210	29 230	29 080
	Elongation in 2 in., per cent (¼-in. Diameter Round Bars)						Elongation in 2 in., per cent (⅛ by ½-in. Flat Bars)				
C............	3.1	2.6	2.8	3.0	3.0	2.9	2.2	2.3	1.0	2.0	2.0
D............	1.2	1.3	1.5	1.5	1.7	1.4	1.4	1.0	1.2	1.0	1.0
G............	1.6	1.5	2.6	1.9	1.7	1.5	1.8	1.5	2.0	1.0	1.4
S............	1.5	1.8	2.0	1.2	2.0	1.3	1.5	1.0	1.0	1.1	0.8
W............	1.2	1.0	1.3	1.1	1.0	0.8	1.0	1.0	1.0	0.9	1.4
Average............	1.7	1.6	2.0	1.7	1.9	1.6	1.6	1.4	1.2	1.2	1.3
	Charpy Impact Value, ft-lb. (¼-in. Square Bars)										
C............	3.5					2.4					
D............	[a]					1.3					
G............	3.0					1.6					
S............	2.3					1.6					
W............	1.7					1.1					
Average............	2.6					1.6					

[a] Test data not available.

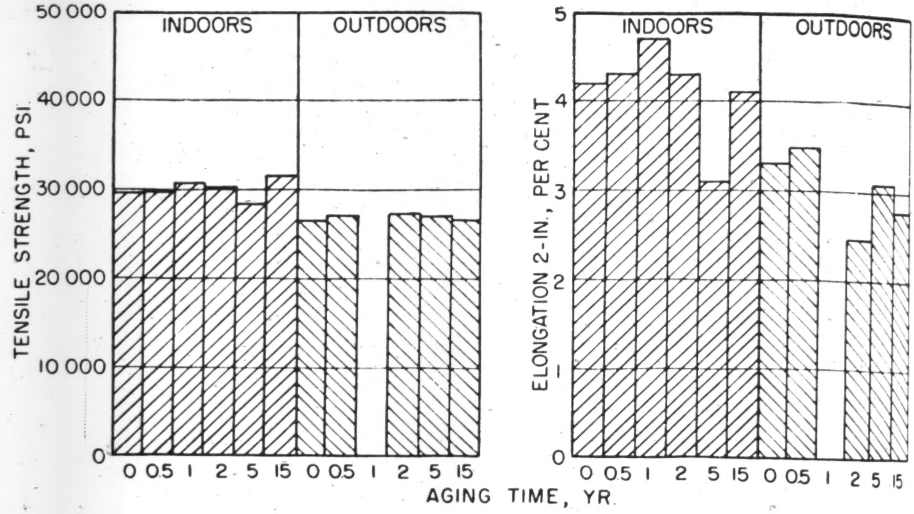

FIG. 10.—Aging Characteristics of Aluminum-Base Alloy No. VIII.

TABLE VIII.—ALUMINUM-BASE ALLOY NO. VIII.

Producer	Copper	Silicon	Nickel	Iron	Manganese	Magnesium	Zinc	Aluminum	Other Elements
	Coordinated Chemical Analyses—All Laboratories, per cent								
Nominal Composition...	1.5	0.75	2.25	1.5 to 2.0	0.05 Max.	0.05 Max.	....	Remainder	....
C...........	1.56	0.46	1.50	1.67	0.02	0.01	0.03	94.75	nil
D...........	1.90	0.85	2.22	1.70	0.05	0.03	0.10	93.15	nil
G...........	1.63	1.43	1.84	1.58	0.10	0.01	nil	93.41	nil
S...........	1.85	0.95	2.38	1.63	0.06	0.03	0.05	93.05	nil
W...........	0.45	2.06	0.90	3.52	[illegible]	0.01	0.07	92.91	nil

Producer	INDOOR EXPOSURE AT NEW YORK, N. Y.						OUTDOOR EXPOSURE AT NEW YORK, N. Y.				
	As Cast	6 Months	1 yr.	2 yr.	5 yr.	15 yr.	As Cast	6 Months	2 yr.	5 yr.	15 yr.
	Tensile Strength, psi. (¼-in. Diameter Round Bars)						Tensile Strength, psi. (⅛ by ½-in. Flat Bars)				
C...........	31 260	31 680	32 880	32 830	[a]	33 850	29 380	27 440	29 560	29 440	28 940
D...........	24 640	28 540	28 340	27 840	27 820	27 210	23 980	27 080	26 170	24 330	24 760
G...........	27 990	25 980	27 680	26 190	26 650	29 900	26 010	25 580	26 510	27 070	26 780
S...........	34 850	33 140	33 770	34 350	35 100	35 760	26 200	27 400	26 740	27 300	26 740
W...........	30 380	30 440	31 350	30 200	25 130	31 220	28 410	28 800	28 850	27 650	27 260
Average...........	29 820	29 960	30 800	30 280	28 680	31 590	26 800	27 260	27 570	27 160	26 900
	Elongation in 2 in., per cent (¼-in. Diameter Round Bars)						Elongation in 2 in., per cent (⅛ by ½-in. Flat Bars)				
C...........	5.8	6.2	6.7	6.5	1.6	5.3	4.3	4.7	2.3	3.0	4.0
D...........	2.5	3.4	3.0	2.9	3.3	2.5	2.1	2.6	1.0	3.0	2.0
G...........	2.9	3.0	3.6	2.9	2.9	3.3	3.0	2.7	2.4	2.5	2.1
S...........	5.0	4.9	5.0	4.8	5.2	4.4	3.0	2.5	2.8	3.0	2.7
W...........	4.7	4.0	5.2	4.3	2.5	4.9	4.1	4.8	4.0	4.0	3.3
Average...........	4.2	4.3	4.7	4.3	3.1	4.1	3.3	3.5	2.5	3.1	2.8
	Charpy Impact Value, ft-lb. (¼-in. Square Bars)										
C...........	5.2					3.5					
D...........	[a]					3.8					
G...........	[a]					3.0					
S...........	5.6					5.0					
W...........	9.4					5.6					
Average...........	6.7					4.2					

[a] Test data not available.

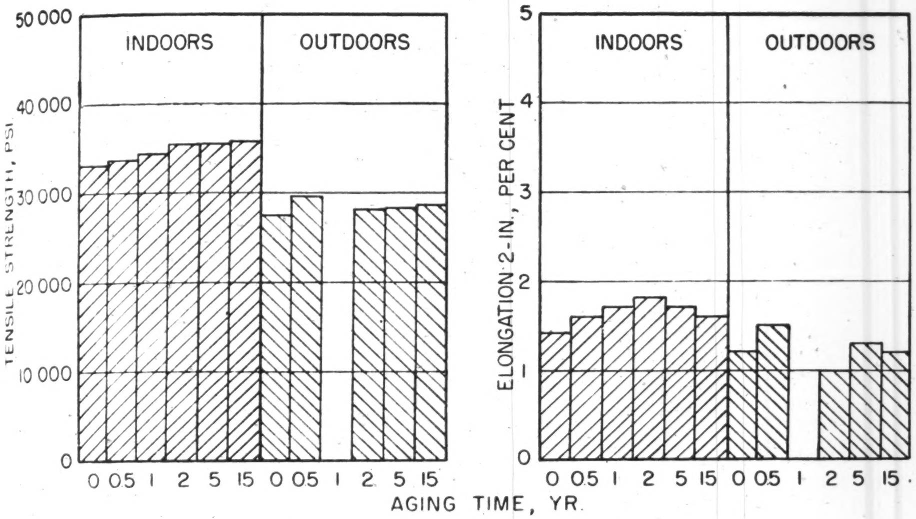

FIG. 11.—Aging Characteristics of Aluminum-Base Alloy No. IX.

TABLE IX.—ALUMINUM-BASE ALLOY NO. IX.

Producer	Coordinated Chemical Analyses—All Laboratories, per cent								
	Copper	Silicon	Nickel	Iron	Manganese	Magnesium	Zinc	Aluminum	Other Elements
Nominal Composition...	4.0	1.5	4.0	1.5 to 2.0	0.05 Max.	0.05 Max.	....	Remainder	....
C..........	4.12	2.30	4.31	1.19	0.63	0.01	0.28	87.16	nil
D..........	3.86	1.64	3.78	1.15	0.04	0.04	0.23	89.26	nil
G..........	4.07	1.46	3.68	1.74	0.03	0.01	nil	89.01	nil
S..........	4.15	1.55	3.83	1.68	0.06	0.02	0.12	88.59	nil
W..........	3.28	3.58	1.06	4.97	0.08	0.01	0.18	86.84	nil

Producer	Indoor Exposure at New York, N. Y.						Outdoor Exposure at New York, N. Y.				
	As Cast	6 Months	1 yr.	2 yr.	5 yr.	15 yr.	As Cast	6 Months	2 yr.	5 yr.	15 yr.
	Tensile Strength, psi. (¼-in. Diameter Round Bars)						Tensile Strength, psi. (⅛ by ½-in. Flat Bars)				
C..........	32 180	31 300	37 560	37 260	35 410	36 430	28 930	32 240	29 650	30 840	29 830
D..........	25 770	29 180	27 840	29 230	29 170	27 970	24 690	27 160	25 090	25 180	23 730
G..........	31 850	31 780	31 500	34 370	35 000	35 060	28 680	29 140	27 860	29 500	28 680
S..........	36 690	36 620	35 590	37 600	38 440	39 120	24 480	27 000	26 130	23 780	29 050
W..........	38 580	38 740	39 640	39 470	40 210	41 360	32 120	33 580	32 910	33 350	33 520
Average..........	33 010	33 520	34 430	35 590	35 650	35 990	27 780	29 820	28 330	28 530	28 960
	Elongation in 2 in., per cent (¼-in. Diameter Round Bars)						Elongation in 2 in., per cent (⅛ by ½-in. Flat Bars)				
C..........	0.7	1.4	1.7	1.0	1.1	1.0	1.0	1.5	0.5	1.0	0.7
D..........	1.0	1.0	1.5	1.5	1.9	2.9	1.0	1.0	1.0	1.5	1.2
G..........	1.8	2.0	1.5	1.8	1.8	1.0	1.6	2.0	1.4	1.2	1.5
S..........	1.8	2.0	1.9	2.5	2.0	1.8	1.0	1.3	1.0	1.1	1.0
W..........	1.5	1.5	1.8	2.0	1.5	1.5	1.2	1.5	1.0	1.5	1.5
Average..........	1.4	1.6	1.7	1.8	1.7	1.6	1.2	1.5	1.0	1.3	1.2
	Charpy Impact Value, ft-lb. (¼-in. Square Bars)										
C..........	1.9					1.1					
D..........	[a]					1.2					
G..........	2.8					1.6					
S..........	2.7					0.6					
W..........	2.6					1.8					
Average..........	2.5					1.3					

[a] Test data not available.

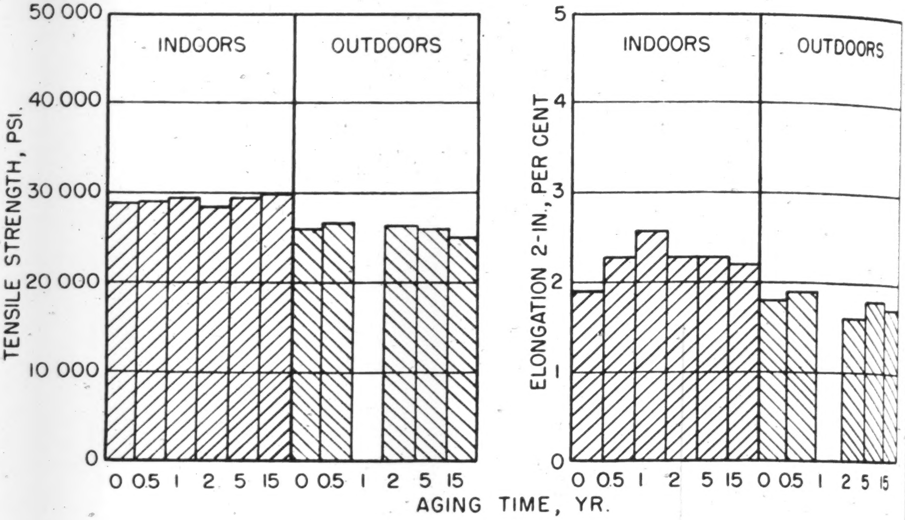

FIG. 12.—Aging Characteristics of Aluminum-Base Alloy No. X.

TABLE X.—ALUMINUM-BASE ALLOY NO. X.

Producer	Coordinated Chemical Analyses—All Laboratories, per cent								
	Copper	Silicon	Nickel	Iron	Manganese	Magnesium	Zinc	Aluminum	Other Elements
Nominal Composition...	....	2.0	5.0	1.5 to 2.0	0.05 Max.	0.05 Max.	....	Remainder	....
C...........	0.44	2.06	4.39	1.05	0.73	0.09	0.09	91.5	nil
D...........	0.20	2.30	4.54	1.00	0.05	0.04	0.27	91.60	nil
G...........	0.52	1.93	4.61	1.47	0.04	0.01	0.08	91.34	nil
S...........	0.32	2.03	4.89	1.63	0.07	0.04	0.13	90.89	nil
W...........	0.76	1.78	4.29	1.88	0.06	Trace	0.24	90.99	nil

Producer	INDOOR EXPOSURE AT NEW YORK, N. Y.						OUTDOOR EXPOSURE AT NEW YORK, N. Y.				
	As Cast	6 Months	1 yr.	2 yr.	5 yr.	15 yr.	As Cast	6 Months	2 yr.	5 yr.	15 yr.
	Tensile Strength, psi. (¼-in. Diameter Round Bars)						Tensile Strength, psi. (⅛ by ½-in. Flat Bars)				
C...........	33 420	31 920	33 720	31 740	32 820	34 660	30 320	31 940	29 010	30 600	29 670
D...........	23 690	23 720	23 700	24 230	22 980	23 000	21 030	21 180	22 770	21 290	18 170
G...........	27 650	28 640	27 980	25 040	29 580	28 930	25 440	25 300	25 700	24 820	25 580
S...........	32 340	32 800	32 250	32 710	32 450	32 630	26 980	28 150	27 020	27 890	26 460
W...........	28 200	28 580	29 990	29 310	30 110	30 720	27 520	27 870	28 150	26 700	26 360
Average...........	29 060	29 130	29 530	28 610	29 590	29 990	26 260	26 890	26 530	26 260	25 250
	Elongation in 2 in., per cent (¼-in. Diameter Round Bars)						Elongation in 2 in., per cent (⅛ by ½-in. Flat Bars)				
C...........	2.8	2.3	2.5	2.8	2.1	2.7	2.3	2.6	1.0	2.0	1.8
D...........	1.7	1.5	2.5	1.7	1.9	2.0	1.7	1.4	1.7	1.5	1.5
G...........	1.8	3.0	2.3	1.6	2.1	2.4	1.9	2.2	1.9	1.6	1.8
S...........	1.9	2.6	2.8	3.1	2.9	2.0	1.5	1.4	1.3	1.5	1.8
W...........	1.5	2.3	2.8	2.3	2.4	2.0	1.7	2.0	2.1	2.5	1.5
Average...........	1.9	2.3	2.6	2.3	2.3	2.2	1.8	1.9	1.6	1.8	1.7
	Charpy Impact Value, ft-lb. (¼-in. Square Bars)										
C...........	3.2					2.3					
D...........	[a]					2.0					
G...........	2.0					2.2					
S...........	3.3					2.9					
W...........	3.2					2.9					
Average...........	2.9					2.5					

[a] Test data not available.

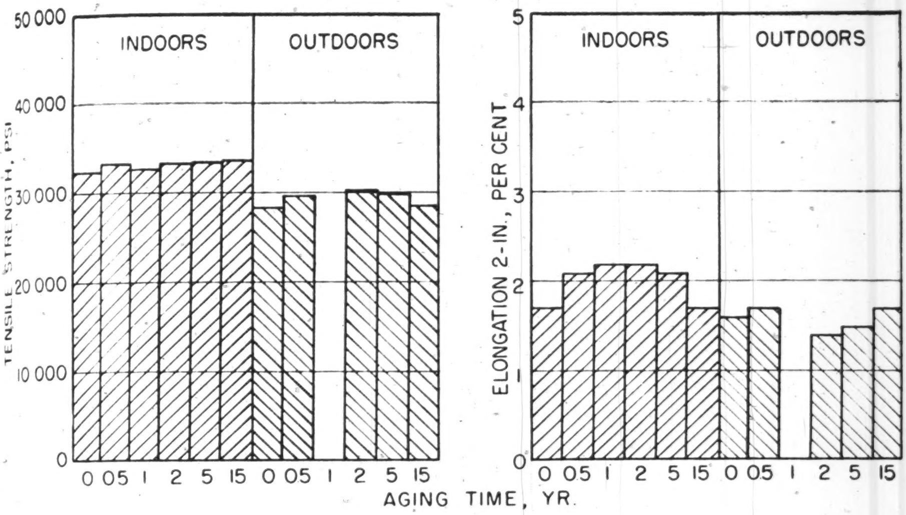

Fig. 13.—Aging Characteristics of Aluminum-Base Alloy No. XI.

TABLE XI.—ALUMINUM-BASE ALLOY NO. XI.

Producer	Coordinated Chemical Analyses—All Laboratories, per cent								
	Copper	Silicon	Nickel	Iron	Manganese	Magnesium	Zinc	Aluminum	Other Elements
Normal Composition	2.0	8.0	....	1.5 to 2.0	0.05 Max.	0.05 Max.	....	Remainder	....
C	2.0	7.48	0.67	0.99	0.70	0.02	0.10	88.04	nil
D	0.33	7.95	nil	1.40	0.04	0.03	0.51	89.74	nil
G	2.09	7.65	nil	0.90	0.03	0.03	0.06	89.08	tin 0.16
S	2.07	7.30	0.21	1.92	0.02	0.04	0.08	88.36	nil
W	1.46	6.86	0.37	2.91	0.04	0.01	0.36	87.99	nil

Producer	Indoor Exposure at New York, N. Y.						Outdoor Exposure at New York, N. Y.				
	As Cast	6 Months	1 yr.	2 yr.	5 yr.	15 yr.	As Cast	6 Months	2 yr.	5 yr.	15 yr.
	Tensile Strength, psi. (¼-in. Diameter Round Bars)						Tensile Strength, psi. (⅛ by ½-in. Flat Bars)				
C	37 690	37 280	36 460	39 410	38 970	38 600	30 210	31 680	30 840	31 840	30 510
D	28 650	30 920	30 560	29 550	28 640	30 780	27 250	28 520	29 440	28 170	28 850
G	31 700	31 540	32 220	31 830	32 900	35 040	31 960	31 160	30 840	31 610	28 290
S	34 440	35 020	34 230	34 650	35 770	35 940	27 540	28 800	28 430	30 040	28 590
W	29 190	30 400	29 800	30 480	32 380	30 270	26 140	29 200	31 200	28 020	28 220
Average	32 330	33 030	32 650	33 180	33 730	34 130	28 620	29 870	30 150	29 940	28 890
	Elongation in 2 in., per cent (¼-in. Diameter Round Bars)						Elongation in 2 in., per cent (⅛ by ½-in. Flat Bars)				
C	2.7	3.1	2.9	2.9	3.1	2.6	2.1	2.0	1.1	1.5	1.5
D	2.0	2.1	2.7	2.4	1.9	2.0	1.7	1.8	1.8	1.8	2.2
G	1.8	2.1	2.1	2.0	2.0	2.0	2.6	2.4	2.0	2.0	2.5
S	1.0	1.8	1.8	2.0	1.9	1.5	1.0	1.1	1.0	1.6	1.0
W	0.9	1.3	1.3	1.6	1.4	0.5	0.5	1.0	1.0	0.5	1.5
Average	1.7	2.1	2.2	2.2	2.1	1.7	1.6	1.7	1.4	1.5	1.7
	Charpy Impact Value, ft-lb. (¼-in. Square Bars)										
C	3.6					2.2					
D	[a]					2.6					
G	3.7					2.0					
S	2.5					2.0					
W	1.6					1.2					
Average	2.8					2.0					

[a] Test data not available.

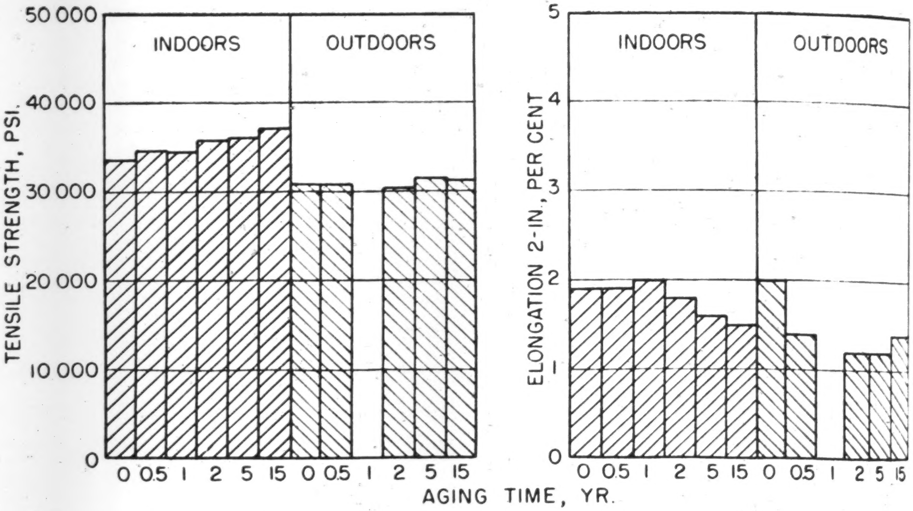

FIG. 14.—Aging Characteristics of Aluminum-Base Alloy No. XII.

TABLE XII.—ALUMINUM-BASE ALLOY NO. XII.

Producer	Copper	Silicon	Nickel	Iron	Manganese	Magnesium	Zinc	Aluminum	Other Elements
	Coordinated Chemical Analyses—All Laboratories, per cent								
Nominal Composition...	8.0	1.0	....	1.5 to 2.0	0.05 Max.	0.05 Max.	....	Remainder	...
C...........	7.62	0.72	0.10	1.74	0.01	0.02	0.04	89.75	nil
D...........	7.86	1.20	0.04	1.60	0.02	0.18	0.14	88.96	nil
G...........	7.90	1.17	nil	1.00	0.02	0.03	0.07	89.41	tin 0.4
S...........	7.55	1.48	nil	1.97	0.01	0.03	0.09	88.87	nil
W...........	6.83	3.08	0.03	2.57	0.05	0.01	0.12	87.31	nil

Producer	INDOOR EXPOSURE AT NEW YORK, N. Y.						OUTDOOR EXPOSURE AT NEW YORK, N. Y.				
	As Cast	6 Months	1 yr.	2 yr.	5 yr.	15 yr.	As Cast	6 Months	2 yr.	5 yr.	15 yr.
	Tensile Strength, psi. (¼-in. Diameter Round Bars)						Tensile Strength, psi. (⅛ by ½-in. Flat Bars)				
C...........	34 880	35 400	36 640	37 830	37 470	39 140	31 140	27 930	28 610	29 590	30 680
D...........	32 380	35 820	35 980	35 830	36 040	34 540	31 950	32 820	28 300	29 780	30 000
G...........	30 840	31 520	26 270	30 390	32 760	33 820	24 630	24 880	25 800	27 090	25 520
S...........	34 670	36 380	36 850	38 930	40 890	39 570	32 910	35 800	36 240	35 940	35 100
W...........	35 640	35 120	37 080	36 390	33 580	38 780	34 170	33 160	32 870	35 040	34 500
Average...........	33 680	34 850	34 560	35 870	36 150	37 170	30 960	30 920	30 360	31 490	31 160
	Elongation in 2 in., per cent (¼-in. Diameter Round Bars)						Elongation in 2 in., per cent (⅛ by ½-in. Flat Bars)				
C...........	2.3	2.8	2.8	2.8	2.1	2.0	2.8	1.8	0.8	1.3	1.5
D...........	1.0	1.5	1.5	1.1	1.3	1.0	1.2	1.3	1.0	0.8	1.3
G...........	1.7	2.0	1.7	1.8	1.5	1.7	1.7	1.4	1.2	1.0	1.5
S...........	2.5	1.9	2.4	1.6	2.3	1.8	2.5	1.5	2.0	1.8	1.3
W...........	2.0	1.5	1.6	1.8	1.0	0.8	2.0	1.0	1.2	1.0	1.5
Average...........	1.9	1.9	2.0	1.8	1.6	1.5	2.0	1.4	1.2	1.2	1.4
	Charpy Impact Value, ft-lb. (¼-in. Square Bars)										
C...........	3.7					2.0					
D...........	[a]					1.7					
G...........	3.9					1.9					
S...........	4.0					2.7					
W...........	2.3					1.5					
Average...........	3.5					2.0					

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[a] Test data not available.

sile strength occurred during the first 2 yr. of the outdoor exposure. The continued loss of elongation after this period of time, noted in the case of the indoor aging tests, was not observed in the outdoor studies.

If consideration is given to the aging of the alloys and the extent of corrosion estimated as the net change in tensile strength occurring between the shelf-life aging and the outdoor exposure tests, all of the aluminum-base alloys showed net losses in tensile strength of 5 to 12 per cent. This conclusion is based on the assumption that the percentage change in tensile strength due to aging was essentially the same for both the round specimens exposed indoors and for the flat specimens exposed outdoors. The percentage change in tensile strength occurring as a result of indoor aging was therefore applied as a correction to the tensile strength observed in the outdoor tests in evaluating the combined effect of aging and corrosion. For example, if the tensile strength of an alloy after 15 yr. of outdoor exposure was 102 per cent of the initial tensile strength, while the gain due to indoor aging was 5 per cent, it was concluded that there had been a net loss of 3 per cent due to corrosion.

Zinc-Base Alloys:

In analyzing the data presented in Tables XIII to XXI and Figs. 15 to 24 for the zinc-base die-casting alloys it is necessary to consider not only the effect of variations in composition on the aging characteristics and corrosion resisting properties, but also the effect of the various grades of zinc used in compounding the different alloys. This consideration is necessary because it has been shown that small percentages of certain impurities, notably lead, tin, and cadmium, have a marked effect upon the aging characteristics of zinc-base alloys. It should be noted that, in the case of some of the zinc alloys, such wide departure from the nominal composition occurred in the case of certain producers as to make the average physical properties of specimens from the several producers questionable except in so far as the results of these tests indicate a broad over-all trend. This we believe, is indicated by the 15-yr. data. To permit evaluation of these trends, the various alloys have been divided into four groups for purposes of analysis. These are:

Group A

Alloy No. XIII: 0.25 per cent aluminum, 5 per cent copper, remainder High-Grade Zinc.

Group B

Alloy No. XVII: 3 per cent aluminum, 3 per cent copper, 0.3 per cent magnesium, remainder High-Grade Zinc.

Group C

Alloy No. XVIII-A: 0.5 per cent aluminum, 3 per cent copper, 6 per cent tin, remainder Prime Western Zinc.

Alloy No. XVIII-B: 0.5 per cent aluminum, 3 per cent copper, 6 per cent tin, remainder High-Grade Zinc.

Group D

Alloy No. XIV: 4 per cent aluminum, 3 per cent copper, remainder High-Grade Zinc.

Alloy No. XX: 4 per cent aluminum, 3 per cent copper, remainder a special grade of zinc (minimum, 99.75 per cent zinc) known as Electro-Engraver's Special.

Alloy No. XV: 4 per cent aluminum, 3 per cent copper, 0.1 per cent magnesium, remainder High-Grade Zinc.

Alloy No. XVI: 4 per cent aluminum, 3 per cent copper, 0.1 per cent magnesium, remainder Special High-Grade Zinc (minimum, 99.94 per cent zinc).

Alloy No. XXI: 4 per cent aluminum, 3 per cent copper, 0.1 per cent magnesium, remainder Special High-Grade Zinc (minimum, 99.98 per cent zinc).

Alloy No. XIX: 4 per cent aluminum, 2 per cent copper, 0.1 per cent magnesium, 0.02 per cent nickel, remainder Special High-Grade Zinc (minimum, 99.94 per cent zinc).

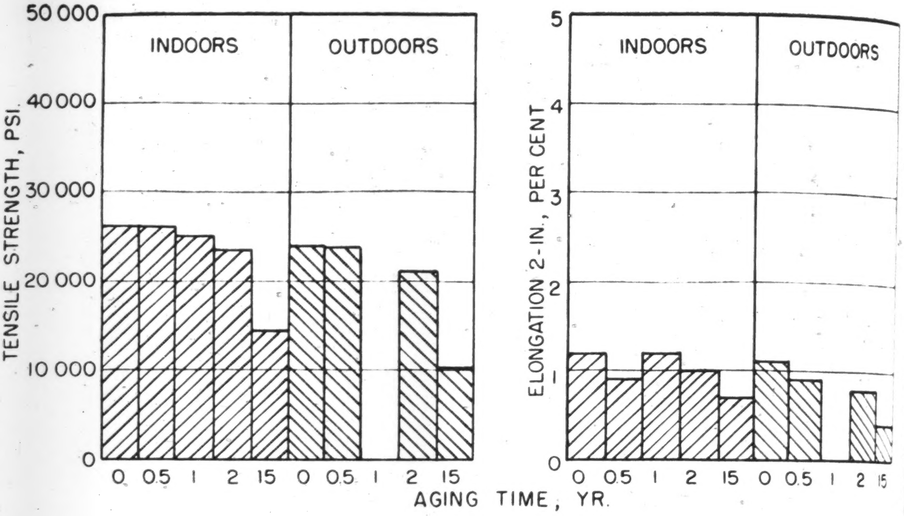

FIG. 15.—Aging Characteristics of Zinc-Base Alloy No. XIII.

TABLE XIII.—ZINC-BASE ALLOY NO. XIII.

Producer	Coordinated Chemical Analyses—All Laboratories, per cent							
	Aluminum	Copper	Magnesium	Lead	Cadmium	Tin	Zinc	Grade of Zinc
Nominal Composition	0.25	5.0	....	....	....	....	Remainder	High Grade
C	0.23	4.90	nil	0.049	0.027	0.03	Remainder	
D	0.33	4.62	0.0032	0.036	0.046	0.027	Remainder	
R	0.25	4.45	0.003	0.042	0.0099	0.044	Remainder	
S	[a]	[a]	[a]	[a]	[a]	[a]	[a]	
T	0.24	5.44	0.0028	0.040	0.079	0.003	Remainder	

Producer	Indoor Exposure at New York, N. Y.					Outdoor Exposure at New York, N. Y.			
	As Cast	6 Months	1 yr.	2 yr.	15 yr.	As Cast	6 Months	2 yr.	15 yr.
	Tensile Strength, psi. (¼-in. Diameter Round Bars)					Tensile Strength, psi. (⅛ by ½-in. Flat Bars)			
C	22 580	23 410	22 230	20 350	21 120	18 600	21 110	20 610	11 320
D	29 730	29 080	26 880	25 660	16 160	25 800	25 660	21 690	13 870
R	24 340	24 510	24 440	21 970	17 040	23 610	23 680	19 510	9 340
S	[a]	28 920	25 410	25 670	14 550	25 260	24 260	22 630	8 840
T	29 580	26 700	26 620	25 710	5 380[b]	27 600	25 640	21 090	7 740
Average	26 560	26 520	25 120	23 870	14 380	24 170	24 070	21 110	10 220
	Elongation in 2 in., per cent (¼-in. Diameter Round Bars)					Elongation in 2 in., per cent (⅛ by ½-in. Flat Bars)			
C	0.5	1.0	0.6	0.9	0.6	0.5	0.5	1.0	0
D	1.6	1.0	1.5	0.8	0.5	1.3	1.0	0.8	1.0
R	1.0	1.2	2.0	1.0	1.0	1.0	0.8	0.5	0.6
S	[a]	1.0	0.6	1.5	0.5	1.0	1.0	1.0	0.5
T	1.5	0.5	1.2	1.0	0.8	1.5	1.0	0.9	0.0
Average	1.2	0.9	1.2	1.0	0.7	1.1	0.9	0.8	0.4
	Charpy Impact Value, ft-lb. (¼-in. Square Bars)								
C	0.75				0.47				
D	1.00				0.30				
R	0.75				0.30				
S	[a]				[c]				
T	1.00				0.30				
Average	0.88				0.30				

[a] Test data not available.
[b] Average of 2 tests. 8 specimens broke in applying gage marks.
[c] No test specimens available.

Group A.—Alloy No. XIII showed about the same loss in tensile strength during shelf-life aging as on outdoor aging through the first 2-yr. exposure, after which the outdoor exposure caused a somewhat greater loss in tensile strength. A similar effect was noted in the elongation. After 15 yr. of indoor aging the impact strength decreased to about one third of the original value and the actual impact strength was very low, being but 0.3 ft-lb.

Group B.—Alloy No. XVII showed somewhat less loss in tensile strength and elongation than alloy No. XIII during both indoor and outdoor aging. It also showed very little difference in the effect of indoor and outdoor aging during the first 2 yr. After 15-yr. exposure, the loss in tensile strength and elongation was somewhat greater for the outdoor than for the indoor aging tests. The loss in impact strength was less than that shown by any other alloy although the actual value of 0.7 ft-lb. is quite low.

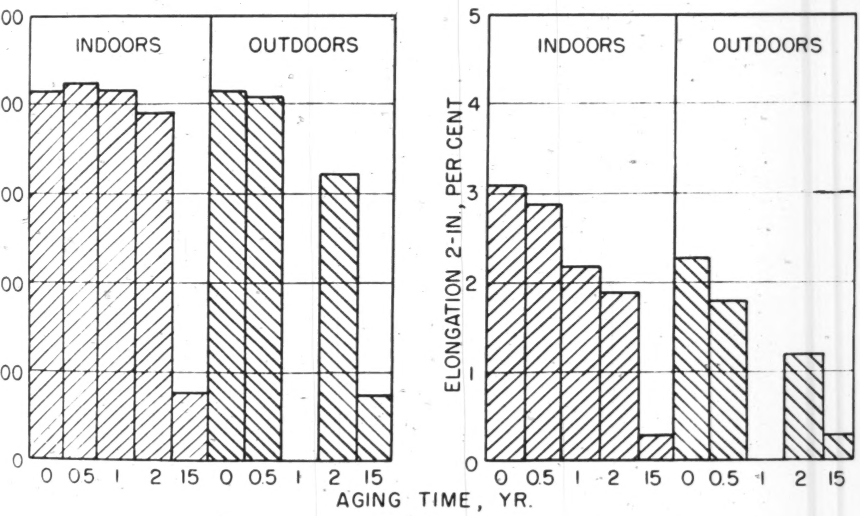

FIG. 16.—Aging Characteristics of Zinc-Base Alloy No. XIV.

Group C.—In the case of alloys Nos. XVIII-A and XVIII-B, those specimens made from High-Grade Zinc showed less loss in tensile strength due to shelf-life aging than did those made from Prime Western Zinc. However, in the outdoor exposure tests there appeared to be no difference in the corrosion resistance of these two alloys. In both alloys the loss in tensile strength was more than 60 per cent. Unfortunately a good comparison is difficult because alloy No. XVIII-B was die-cast by only one producer as compared to alloy No. XVIII-A which was die-cast by four producers. Impact data are lacking for alloy No. XVIII-B, but in the case of alloy No. XVIII-A the impact strength decreased by more than 75 per cent during 15 yr. of indoor aging.

Group D.—Alloys Nos. XIV, and XX, which have similar compositions except that the former was made from High-Grade Zinc and the latter from Electro-Engraver's Special Zinc (minimum, 99.75 per cent zinc), showed marked differences in their aging characteristics. The alloy made from the High-Grade Zinc showed greater loss in tensile strength, both on indoor and outdoor aging, than did the alloy made from the

Electro-Engraver's Special Zinc. The effect of the basis metal on elongation was equally marked, but this loss was not so evident as was the loss of impact strength on indoor aging. Both alloys have impact strengths of only 0.3 ft-lb. after 15 yr.

TABLE XIV.—ZINC-BASE ALLOY NO. XIV.

Producer	Coordinated Chemical Analyses—All Laboratories, per cent							
	Aluminum	Copper	Magnesium	Lead	Cadmium	Tin	Zinc	Grade of Zinc
Nominal Composition	4.0	3.0	....	....	....	....	Remainder	High Grade
C	3.37	3.48	nil	0.035	0.037	nil	Remainder	
D	3.87	3.21	0.0024	0.035	0.035	0.029	Remainder	
R	3.79	4.45	0.0049	0.034	0.012	0.041	Remainder	
S	4.21	3.81	0.0050	0.044	0.027	0.003	Remainder	
T	3.83	2.86	0.0017	0.040	0.002	0.011	Remainder	
Z	[a]	[a]	[a]	[a]	[a]	[a]	[a]	

Producer	INDOOR EXPOSURE AT NEW YORK, N. Y.					OUTDOOR EXPOSURE AT NEW YORK, N. Y.			
	As Cast	6 Months	1 yr.	2 yr.	15 yr.	As Cast	6 Months	2 yr.	15 yr.
	Tensile Strength, psi. (¼-in. Diameter Round Bars)					Tensile Strength, psi. (⅛ by ½-in. Flat Bars)			
C	44 260	43 810	44 450	43 600	17 630[c]	42 180	40 100	33 430	10 440
D	41 750	43 180	43 640	39 710	520[d]	41 990	41 510	33 160	9 620
R	41 710	38 640	39 910	33 610	0[e]	42 340	38 710	26 520	420[f]
S	[a]	44 620	43 730	43 910	11 420	43 940	43 250	35 190	11 230
T	40 850	42 880	38 920	36 570	6 840	40 040	43 710	31 800	7 730
Z	38 030	40 480	38 110	36 970	11 370	38 870	37 920	32 660	5 550
Average	41 320	42 270	41 460	39 060	7 960	41 560	40 870	32 130	7 500
	Elongation in 2 in., per cent (¼-in. Diameter Round Bars)					Elongation in 2 in., per cent (⅛ by ½-in. Flat Bars)			
C	3.5	3.2	3.0	2.5	0.5	1.9	1.0	1.0	0.0
D	3.5	1.3	2.0	1.0	0.0	1.4	1.0	0.5	0.5
R	1.0	1.5	1.0	1.0	0.0	1.5	1.0	0.5	0.0
S	[a]	2.8	2.1	2.9	0.5	2.6	2.1	0.8	0.4
T	3.8	4.4	2.5	1.7	0.1	3.0	2.5	1.6	0.0
Z	3.5	4.2	2.3	2.0	0.5	3.5	3.1	2.7	1.0
Average	3.1	2.9	2.2	1.9	0.3	2.3	1.8	1.2	0.3
	Charpy Impact Value, ft-lb. (¼-in. Square Bars)								
C	2.75				0.24				
D	3.25				[b]				
R	2.25				0.20				
S	5.00				0.40				
T	7.00				0.30				
Z	[a]				[b]				
Average	4.05				0.28				

[a] Test data not available.
[b] No test specimens available.
[c] Only one specimen could be tested. Others fell apart in shelf-life storage.
[d] Average of 6 specimens. 4 specimens broke before testing.
[e] All specimens broke as gage marks were put on them.
[f] Only one specimen tested. Others fell apart on roof-exposure racks.

When 0.1 per cent of magnesium was added to an alloy having otherwise the same composition as that of alloy No. XIV, there was less loss in tensile strength or elongation, both in the indoor and outdoor exposure tests. Whereas on shelf-life aging, the magnesium-free

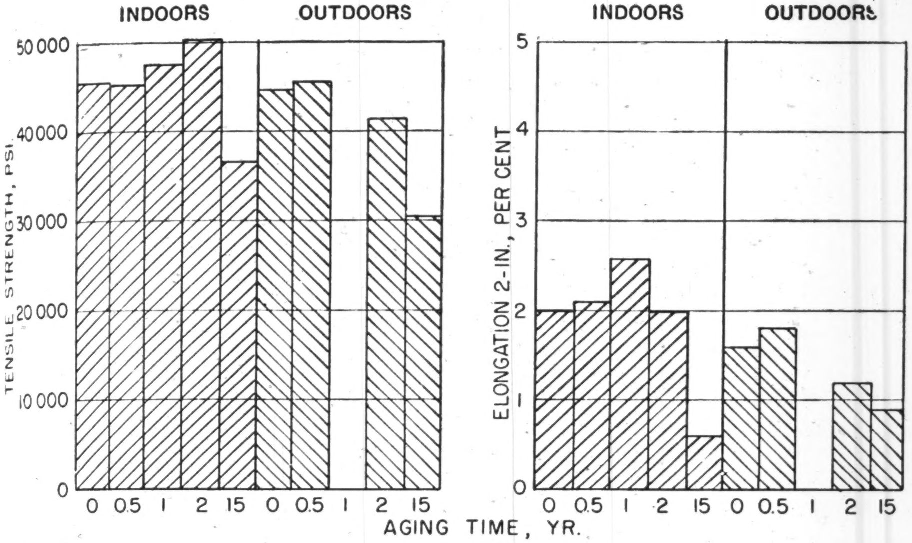

FIG. 17.—Aging Characteristics of Zinc-Base Alloy No. XV.

TABLE XV.—ZINC-BASE ALLOY NO. XV.

Producer	Coordinated Chemical Analyses—All Laboratories, per cent							
	Aluminum	Copper	Magnesium	Lead	Cadmium	Tin	Zinc	Grade of Zinc
Nominal Composition	4.0	3.0	0.1	....	....	....	Remainder	High Grade
C	3.96	3.00	0.10	0.032	0.013	nil	Remainder	
D	4.02	2.97	0.10	0.027	0.051	0.005	Remainder	
R	3.83	3.07	0.07	0.032	0.012	0.009	Remainder	
S	4.04	2.75	0.14	0.041	0.024	0.016	Remainder	
T	[a]	[a]	[a]	[a]	[a]	[a]	[a]	
Z	3.87	3.11	0.11	0.060	0.025	0.0007	Remainder	

Producer	Indoor Exposure at New York, N. Y.					Outdoor Exposure at New York, N. Y.			
	As Cast	6 Months	1 yr.	2 yr.	15 yr.	As Cast	6 Months	2 yr.	15 yr.
	Tensile Strength, psi. (¼-in. Diameter Round Bars)					Tensile Strength, psi. (⅛ by ½-in. Flat Bars)			
C	46 280	47 220	51 500	55 180	50 120	43 680	49 130	40 500	34 850
D	46 830	43 770	47 680	47 660	[b]	45 940	44 310	42 250	27 760
R	51 450	49 600	52 290	55 550	41 000	51 630	49 680	46 350	38 910
S	[a]	45 670	46 870	48 430	34 670	45 490	44 340	42 010	34 160
T	43 120	42 240	45 040	48 530	30 520	45 240	42 210	39 070	27 860
Z	40 270	42 860	42 170	45 330	27 510	36 460	43 520	37 180	18 340
Average	45 590	45 230	47 590	50 110	36 760	44 740	45 530	41 230	30 330
	Elongation in 2 in., per cent (¼-in. Diameter Round Bars)					Elongation in 2 in., per cent (⅛ by ½-in. Flat Bars)			
C	3.3	3.8	5.0	3.4	1.1	2.1	2.1	1.1	1.0
D	1.6	1.7	2.5	1.5	[b]	1.5	0.7	1.1	1.0
R	1.0	1.5	2.0	1.9	1.0	1.0	2.1	0.6	0.0
S	[a]	2.2	2.5	2.0	1.0	1.8	2.0	1.8	0.9
T	1.6	0.9	1.0	1.0	0.5	1.9	2.0	1.6	0.0
Z	2.4	2.5	2.3	2.2	0.5	1.3	2.0	1.0	1.0
Average	2.0	2.1	2.6	2.0	0.8	1.6	1.8	1.2	0.6
	Charpy Impact Value, ft-lb. (¼-in. Square Bars)								
C	4.75				0.93				
D	3.25				[b]				
R	7.00				0.60				
S	4.75				0.60				
T	[a]				[b]				
Z	3.00				0.40				
Average	4.55				0.63				

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[a] Test data not available.

[b] No test specimens available.

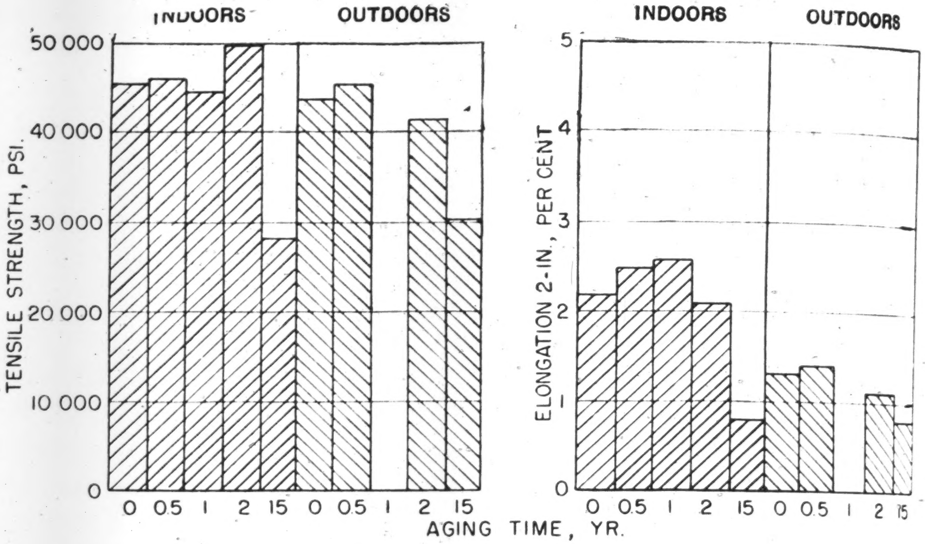

FIG. 18.—Aging Characteristics of Zinc-Base Alloy No. XVI.

TABLE XVI.—ZINC-BASE ALLOY NO. XVI.

Producer	Coordinated Chemical Analyses—All Laboratories, per cent							
	Aluminum	Copper	Magnesium	Lead	Cadmium	Tin	Zinc	Grade of Zinc
Nominal Composition	4.0	3.0	0.1	....	....	....	Remainder	Special High Grade[c] (99.94 per cent Zn)
C	4.26	3.07	0.11	0.037	0.005	....	Remainder	
D	3.99	3.02	0.09	0.045	0.0059	0.004	Remainder	
S	4.08	2.82	0.13	0.038	0.0041	0.016	Remainder	
T	[a]	[a]	[a]	[a]	[a]	[a]	[a]	
Z	3.77	3.04	0.11	0.038	0.002	0.0008	Remainder	

Producer	Indoor Exposure at New York, N. Y.					Outdoor Exposure at New York, N. Y.			
	As Cast	6 Months	1 yr.	2 yr.	15 yr.	As Cast	6 Months	2 yr.	15 yr.
	Tensile Strength, psi. (¼-in. Diameter Round Bars)					Tensile Strength, psi. (⅛ by ½-in. Flat Bars)			
C	49 060	47 640	48 260	54 760	46 220	42 590	47 490	39 390	29 050
D	48 810	46 210	47 180	51 710	[b]	46 530	48 400	45 300	32 640
S	[a]	44 960	44 200	46 550	33 560	43 800	44 110	40 120	28 130
T	44 910	45 460	43 260	48 660	30 720	45 280	46 430	42 910	31 380
Z	40 090	[a]	41 510	47 750	42 930	40 590	42 300	39 650	29 490
Average	45 720	46 070	44 880	49 890	38 360	43 760	45 750	41 470	30 140
	Elongation in 2 in., per cent (¼-in. Diameter Round Bars)					Elongation in 2 in., per cent (⅛ by ½-in. Flat Bars)			
C	3.4	3.6	4.4	2.7	1.0	0.5	1.0	0.8	1.0
D	2.2	2.2	2.5	1.5	[b]	1.2	1.0	1.0	1.0
S	[a]	2.5	2.3	2.5	1.0	1.8	1.9	1.0	1.0
T	1.2	1.9	1.3	1.1	0.5	1.6	1.7	1.6	0.0
Z	2.1	[a]	2.3	2.5	0.5	1.5	1.5	1.1	1.0
Average	2.2	2.5	2.6	2.1	0.8	1.3	1.4	1.1	0.8
	Charpy Impact Value, ft-lb. (¼-in. Square Bars)								
C	4.75				0.91				
D	5.00				[b]				
S	6.00				2.30				
T	[a]				[b]				
Z	5.50				3.30				
Average	5.31				1.84				

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[a] Test data not available.
[b] No test specimens available.
[c] Lead plus cadmium, 0.06 per cent maximum, tin, none, zinc, 99.94 per cent minimum.

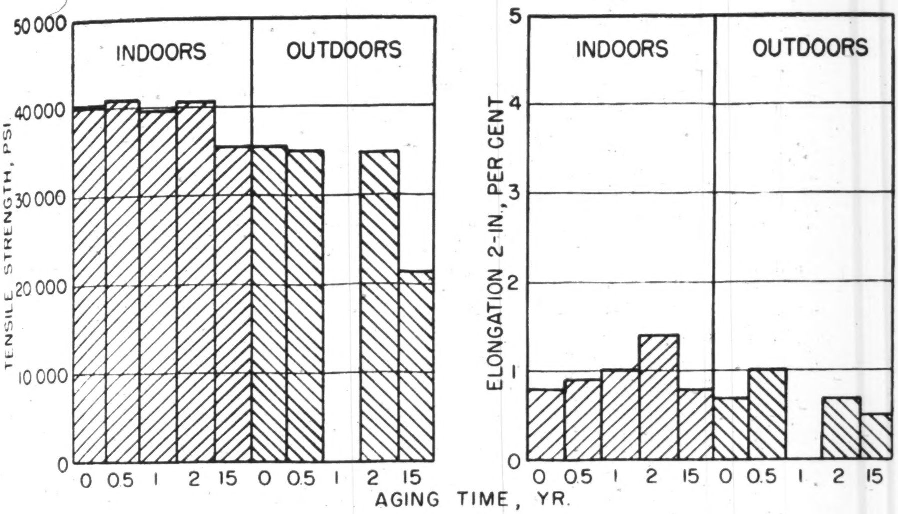

Fig. 19.—Aging Characteristics of Zinc-Base Alloy No. XVII.

TABLE XVII.—ZINC-BASE ALLOY NO. XVII.

Producer	Coordinated Chemical Analyses—All Laboratories, per cent							
	Aluminum	Copper	Magnesium	Lead	Cadmium	Tin	Zinc	Grade of Zinc
Nominal Composition	3.0	3.0	0.3	....	....	....	Remainder	High Grade
C	[a]	[a]	[a]	[a]	[a]	[a]	[a]	
D	3.07	2.94	0.31	0.027	0.043	0.002	Remainder	
R	[a]	[a]	[a]	[a]	[a]	[a]	[a]	
S	3.27	2.60	0.26	0.048	0.019	0.003	Remainder	
T	3.05	2.84	0.33	0.038	0.004	0.001	Remainder	

Producer	Indoor Exposure at New York, N. Y.					Outdoor Exposure at New York, N. Y.			
	As Cast	6 Months	1 yr.	2 yr.	15 yr.	As Cast	6 Months	2 yr.	15 yr.
	Tensile Strength, psi. (¼-in. Diameter Round Bars)					Tensile Strength, psi. (⅛ by ½-in. Flat Bars)			
C	40 920	46 740	41 280	42 490	42 230	30 900	32 270	31 450	19 920
D	39 430	37 040	36 510	36 090	[b]	35 590	34 930	35 750	24 110
R	40 720	38 450	38 890	41 220	35 610	40 760	40 470	37 230	23 570
S	[a]	42 140	42 320	42 390	31 800	36 140	33 030	33 020	22 280
T	40 270	39 630	39 060	40 500	31 670	32 470	33 730	35 920	16 670
Average	40 340	40 800	39 610	40 540	35 330	35 170	34 890	34 670	21 310
	Elongation in 2 in., per cent (¼-in. Diameter Round Bars)					Elongation in 2 in., per cent (⅛ by ½-in. Flat Bars)			
C	1.0	1.1	1.0	2.0	0.5	1.0	1.0	0.7	1.0
D	0.7	0.8	1.0	1.0	[b]	0.5	1.0	0.5	0.5
R	0.5	1.5	1.0	1.0	1.0	0.5	1.0	0.5	0.0
S	[a]	0.4	1.0	1.8	1.0	0.6	1.0	0.7	1.0
T	1.0	0.9	1.0	1.0	0.5	1.0	1.0	0.9	0.0
Average	0.8	0.9	1.0	1.4	0.8	0.7	1.0	0.7	0.5
	Charpy Impact Value, ft-lb. (¼-in. Square Bars)								
C	[a]				0.73				
D	1.50				0.50				
R	[a]				[b]				
S	2.25				1.20				
T	1.50				0.50				
Average	1.75				0.73				

[a] Test data not available.

[b] No test specimens available.

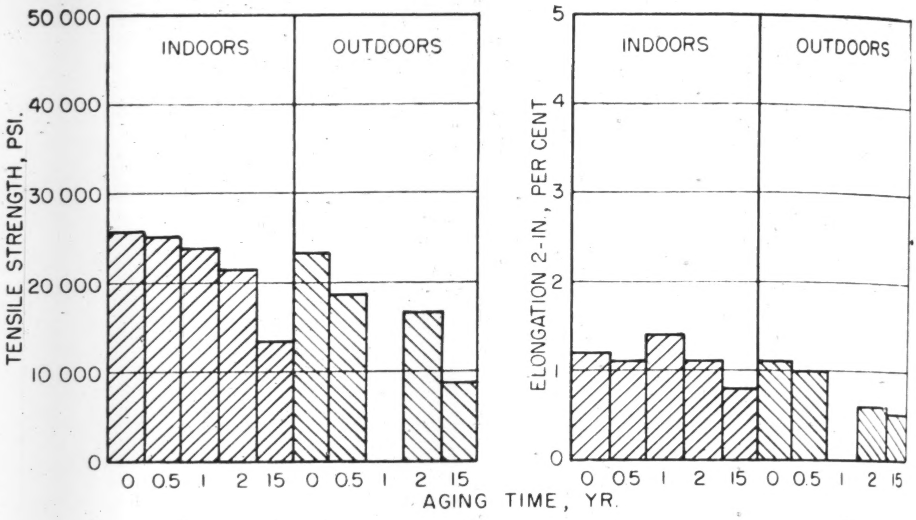

FIG. 20.—Aging Characteristics of Zinc-Base Alloy No. XVIII-A.

TABLE XVIII-A.—ZINC-BASE ALLOY NO. XVIII-A.

Producer	Coördinated Chemical Analyses—All Laboratories, per cent							
	Aluminum	Copper	Magnesium	Lead	Cadmium	Tin	Zinc	Grade of Zinc
Nominal Composition	0.5	3.0	....	....	....	6.0	Remainder	Prime Western
C	0.27	2.82	nil	0.64	0.36	6.67	Remainder	
D	[a]	[a]	[a]	[a]	[a]	[a]	[a]	
R	[a]	[a]	[a]	[a]	[a]	[a]	[a]	
S	0.76	3.34	0.0024	1.05	0.43	5.67	Remainder	
T	0.32	2.72	0.0076	0.49	0.20	5.93	Remainder	

Producer	INDOOR EXPOSURE AT NEW YORK, N. Y.					OUTDOOR EXPOSURE AT NEW YORK, N. Y.			
	As Cast	6 Months	1 yr.	2 yr.	15 yr.	As Cast	6 Months	2 yr.	15 yr.
	Tensile Strength, psi. (¼-in. Diameter Round Bars)					Tensile Strength, psi. (⅛ by ½-in. Flat Bars)			
C	24 480	24 720	25 150	23 560	15 510	21 060	18 530	17 860	8 890
D	26 490	24 750	23 200	24 840	[b]	24 990	21 810	17 560	10 510
R	26 940	27 440	26 710	24 720	15 290	23 780	21 500	17 440	7 630
S	[a]	[a]	20 420	10 530	11 740	[a]	21 120	18 000	9 360
T	26 070	25 470	25 400	24 630	12 170	24 540	11 690	13 990	8 800
Average	26 000	25 580	24 180	21 660	13 680	23 770	18 930	16 970	9 040
	Elongation in 2 in., per cent (¼-in. Diameter Round Bars)					Elongation in 2 in., per cent (⅛ by ½-in. Flat Bars)			
C	1.0	1.5	1.3	1.0	0.7	1.0	1.0	0.5	1.0
D	1.0	1.4	1.3	1.4	[b]	1.0	0.5	0.5	0.3
R	1.5	1.0	2.5	1.0	1.0	1.5	1.4	0.5	0.4
S	[a]	[a]	1.0	0.8	1.0	[a]	1.0	0.5	1.0
T	1.5	0.5	1.0	1.5	0.5	1.2	1.0	1.0	0.0
Average	1.2	1.1	1.4	1.1	0.8	1.2	1.0	0.6	0.5
	Charpy Impact Value, ft-lb. (¼-in. Square Bars)								
C	1.50				0.43				
D	[a]				[b]				
R	[a]				[b]				
S	2.00				0.50				
T	1.50				0.30				
Average	1.67				0.41				

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[a] Test data not available.

[b] No test specimen available.

alloy (No. XIV) had lost approximately 80 per cent of its tensile strength and about 90 per cent of its elongation, the alloy containing magnesium (alloy No. XV) had lost less than 20 per cent of its tensile strength and only 60 per cent of its elongation. In outdoor aging the differences between these two alloys was still evident although it was not quite so great.

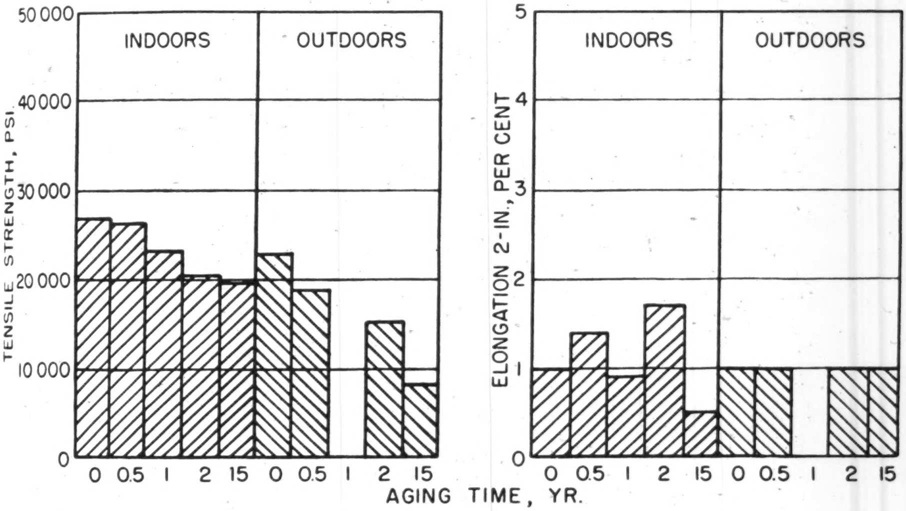

FIG. 21.—Aging Characteristics of Zinc-Base Alloy No. XVIII-B.

TABLE XVIII-B.—ZINC-BASE ALLOY NO. XVIII-B.

Producer	Coordinated Chemical Analyses—All Laboratories, per cent							
	Aluminum	Copper	Magnesium	Lead	Cadmium	Tin	Zinc	Grade of Zinc
Nominal Composition	0.5	3.0	....	....	....	6.0	Remainder	High Grade
C	0.35	2.77	nil	0.037	0.002	5.88	Remainder	

Producer	INDOOR EXPOSURE AT NEW YORK, N. Y.					OUTDOOR EXPOSURE AT NEW YORK, N. Y.			
	As Cast	6 Months	1 yr.	2 yr.	15 yr.	As Cast	6 Months	2 yr.	15 yr.
	Tensile Strength, psi. (¼-in. Diameter Round Bars)					Tensile Strength, psi. (⅛ by ½-in. Flat Bars)			
C	27 180	26 500	23 580	21 510	19 770	23 200	18 960	15 400	8 570
	Elongation in 2 in., per cent (¼-in. Diameter Round Bars)					Elongation in 2 in., per cent (⅛ by ½-in. Flat Bars)			
C	1.0	1.4	0.9	1.7	0.5	1.0	1.0	1.0	1.0

A study of the data obtained for alloys Nos. XVI and XV shows that the use of Special High-Grade Zinc (containing a minimum of 99.94 per cent zinc) in place of High-Grade Zinc as the basis metal in the magnesium-containing alloy had but little effect on either the shelf-life aging or the outdoor aging characteristics. There was, however a slight gain when Special High-Grade Zinc (containing a

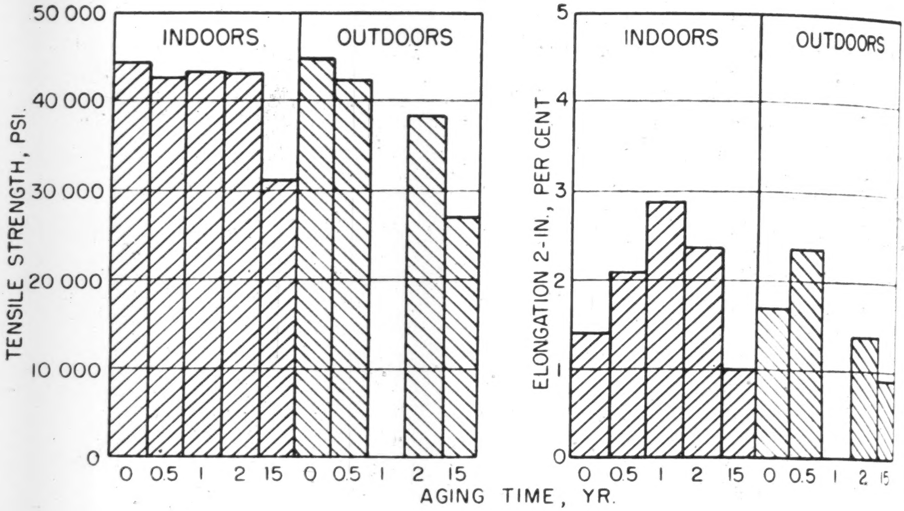

FIG. 22.—Aging Characteristics of Zinc-Base Alloy No. XIX.

TABLE XIX.—ZINC-BASE ALLOY NO. XIX.

Producer	Coordinated Chemical Analyses—All Laboratories, per cent								
	Aluminum	Copper	Magnesium	Lead	Cadmium	Tin	Zinc	Nickel	Grade of Zinc
Nominal Composition	4.0	2.0	0.1	....	....	....	Remainder	0.02	Special High Grade[c] (99.94 per cent Zn)
C	[a]	[a]	[a]	[a]	[a]	[a]	[a]	[a]	
D	4.18	2.19	0.10	0.048	0.005	0.001	Remainder	0.022	
R	3.70	1.94	0.08	0.041	0.011	0.006	Remainder	0.013	
Z	4.00	2.01	0.11	0.035	0.004	0.0007	Remainder	0.030	

Producer	Indoor Exposure at New York, N. Y.					Outdoor Exposure at New York, N. Y.			
	As Cast	6 Months	1 yr.	2 yr.	15 yr.	As Cast	6 Months	2 yr.	15 yr.
	Tensile Strength, psi. (¼-in. Diameter Round Bars)					Tensile Strength, psi. (⅛ by ½-in. Flat Bars)			
D	43 800	41 260	42 400	43 910	[b]	45 020	41 300	39 120	29 030
R	45 030	43 720	43 790	43 090	32 120	46 780	43 850	39 850	27 610
S	[a]	43 110	43 590	42 280	30 640	42 990	41 800	36 220	25 240
Z	[b]	[b]	[b]	[b]	[b]	[b]	[b]	[b]	[b]
Average	44 420	42 700	43 260	43 090	31 380	44 930	42 320	38 400	27 290
	Elongation in 2 in., per cent (¼-in. Diameter Round Bars)					Elongation in 2 in., per cent (⅛ by ½-n. Flat Bars)			
D	1.7	2.3	3.6	2.7	[b]	1.4	3.0	1.9	1.0
R	1.0	2.0	2.5	2.0	1.0	1.5	2.2	0.8	0.6
S	[a]	1.9	2.5	2.5	1.0	2.1	1.9	1.6	1.0
Z	[b]	[b]	[b]	[b]	[b]	[b]	[b]	[b]	[b]
Average	1.4	2.1	2.9	2.4	1.0	1.7	2.4	1.4	0.9
	Charpy Impact Value, ft-lb. (¼-in. Square Bars)								
D	6.00				[b]				
R	5.00				0.60				
Z	5.50				[b]				
Average	5.50				0.60				

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[a] Test data not available.
[b] No test specimens available.
[c] Lead plus cadmium, 0.06 per cent maximum, zinc, 99.94 per cent minimum, nickel, 0.02 per cent nominal.

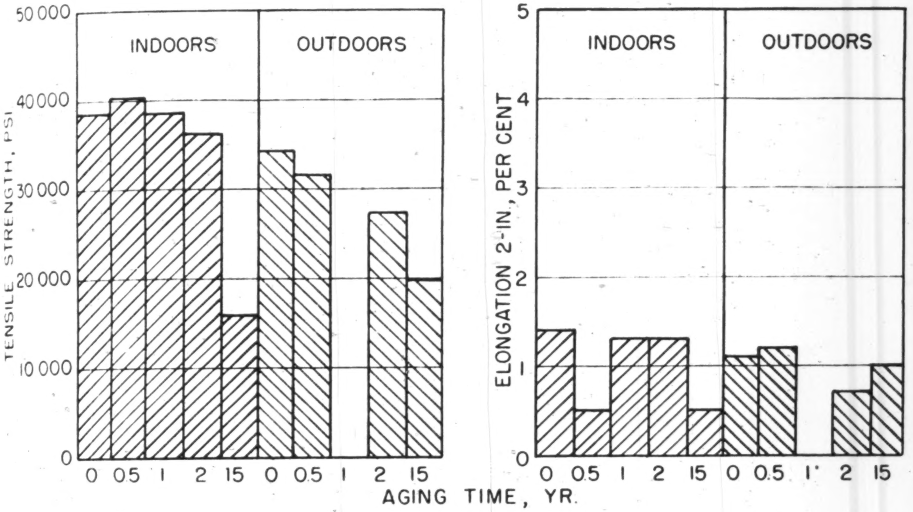

FIG. 23.—Aging Characteristics of Zinc-Base Alloy No. XX.

TABLE XX.—ZINC-BASE ALLOY NO. XX.

Producer	Coordinated Chemical Analyses—All Laboratories, per cent							
	Aluminum	Copper	Magnesium	Lead	Cadmium	Tin	Zinc	Grade of Zinc
Nominal Composition	4.0	3.0	....	....	....	....	Remainder	Special Grade (99.75 per cent Zn) (Electro-Engraver's Special)
C	4.09	2.73	nil	0.027	0.32	0.01	Remainder	
D	3.88	3.14	0.0022	0.24	0.030	0.14	Remainder	

Producer	INDOOR EXPOSURE AT NEW YORK, N. Y.					OUTDOOR EXPOSURE AT NEW YORK, N. Y.			
	As Cast	6 Months	1 yr.	2 yr.	15 yr.	As Cast	6 Months	2 yr.	15 yr.
	Tensile Strength, psi. (¼-in. Diameter Round Bars)					Tensile Strength, psi. (⅛ by ½-in. Flat Bars)			
C	35 710	[a]	35 920	36 140	16 030[c]	30 640	25 740	27 060	17 130[c]
D	41 200	40 330	41 260	36 420	[b]	37 740	37 480	27 740	22 670[d]
Average	38 460	40 330	38 590	36 280	16 030	34 190	31 610	27 400	19 900
	Elongation in 2 in., per cent (¼-in. Diameter Round Bars)					Elongation in 2 in., per cent (⅛ by ½-in. Flat Bars)			
C	1.7	[a]	1.1	1.5	0.5	1.1	1.4	0.8	1.0
D	1.0	0.5	1.5	1.0	[b]	1.1	1.0	0.6	1.0
Average	1.4	0.5	1.3	1.3	0.5	1.1	1.2	0.7	1.0
	Charpy Impact Value, ft-lb. (¼-in. Square Bars)								
C	[a]				0.28				
D	2.25				[b]				
Average	2.25				0.28				

[a] Test data not available.
[b] Test specimens not available.
[c] Average of 9 specimens.
[d] Average of 5 specimens. Others broke on racks.

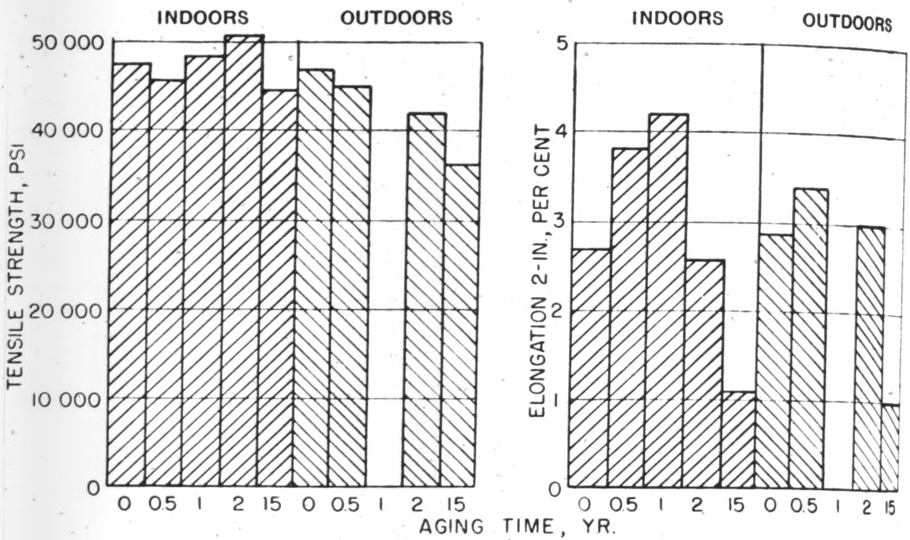

FIG. 24.—Aging Characteristics of Zinc-Base Alloy No. XXI.

TABLE XXI.—ZINC-BASE ALLOY NO. XXI.

Producer	Coordinated Chemical Analyses—All Laboratories, per cent							
	Aluminum	Copper	Magnesium	Lead	Cadmium	Tin	Zinc	Grade of Zinc
Nominal Composition	4.0	3.0	0.1	....	....	....	Remainder	Special High Grade (99.98 per cent Zn)[b]
D	3.93	2.95	0.12	0.0032	0.0067	0.002	Remainder	
R	3.80	2.94	0.09	0.0023	0.0027	0.006	Remainder	
S	3.86	2.92	0.12	0.007	0.0029	0.002	Remainder	
T	4.00	2.93	0.12	0.0025	0.0020	0.0008	Remainder	
Z	4.03	3.01	0.11	0.0030	0.0012	nil	Remainder	

Producer	INDOOR EXPOSURE AT NEW YORK, N. Y.					OUTDOOR EXPOSURE AT NEW YORK, N. Y.			
	As Cast	6 Months	1 yr.	2 yr.	15 yr.	As Cast	6 Months	2 yr.	15 yr.
	Tensile Strength, psi. (¼-in. Diameter Round Bars)					Tensile Strength, psi. (⅛ by ½-in. Flat Bars)			
D	45 470	47 090	47 450	51 130	[a]	45 720	45 820	44 790	34 430
R	51 510	48 630	51 780	55 360	47 800	51 180	49 710	48 390	39 230
S	46 900	46 820	47 920	50 060	51 160	43 690	41 760	39 130	31 670
T	46 940	43 600	47 250	50 250	34 150	48 200	46 380	45 070	39 330
Z	45 280	41 340	47 260	47 610	44 640	44 800	41 680	32 380	37 550
Average	47 220	45 500	48 330	50 880	44 440	46 720	45 070	41 950	36 440
	Elongation in 2 in., per cent (¼-in. Diameter Round Bars)					Elongation in 2 in., per cent (⅛ by ½-in. Flat Bars)			
D	2.1	2.9	2.4	1.0	[a]	3.8	3.3	2.8	1.0
R	2.6	4.1	3.8	2.8	1.0	2.0	3.1	1.2	0.5
S	2.6	3.6	5.1	4.3	2.0	1.7	1.5	1.2	0.5
T	3.2	5.9	5.6	2.7	1.5	3.9	5.0	7.8	1.5
Z	2.8	2.6	3.9	2.0	1.1	2.9	4.1	2.0	1.5
Average	2.7	3.8	4.2	2.6	1.1	2.9	3.4	3.0	1.0
	Charpy Impact Value, ft-lb. (¼-in. Square Bars)								
D	8.25				[a]				
R	8.00				1.40				
S	9.00				1.80				
T	5.75				[a]				
Z	6.00				1.20				
Average	7.40				1.50				

[a] No test specimens available.
[b] Lead plus cadmium, 0.01 per cent maximum, tin, none, zinc, 99.98 per cent minimum.

minimum of 99.98 per cent zinc) was used in compounding this alloy as is shown by the somewhat better aging characteristics of alloy No. XXI as compared to those of alloy No. XVI. In both cases the actual impact strength was better than that of any of the other alloys after shelf-life aging and the tensile strengths were likewise fairly high. There was little difference in the effect of aging on the elongation of these three alloys.

TABLE XXII.—SUMMARY OF METEOROLOGICAL DATA.

Mean annual temperature, deg. Fahr.	52
Number of days per year	
Maximum temperature was:	
32 F. and below	24
90 F. and above	6
Minimum temperature was:	
32 F. and below	93
0 F. and below	1
Precipitation, in.	
Avearge yearly precipitation	42.7
Highest yearly precipitation	58.7
Lowest yearly precipitation	33.2
Average number of days per year of 0.01 in. or over	126
Snowfall, inches per year (melting method)	32.7
Average relative humidity per year, per cent:	
Morning	74
Noon	59
Evening	68
Sunshine:	
Average yearly total, hr.	2680
Percentage of possible hours	60
Wind:	
Average hourly velocity, miles per hour	15
Prevailing direction	Northwest

Alloy No. XIX might be considered as a modification of alloy No. XVI with 0.02 per cent nickel replacing 1 per cent of copper. The changes in tensile strength and elongation for this alloy were comparable to those shown by alloy No. XVI. The loss of impact strength was considerably greater for alloy No. XIX than for alloy No. XVI after 15 yr. of indoor aging.

All of the zinc-base alloys showed losses in tensile strength after 15 yr. of indoor aging. These losses varied from more than 80 per cent for the poorest alloy (No. XIV) to 6 per cent for the best alloy (No. XXI). After 15 yr. of outdoor aging the losses in tensile strength ranged from more than 80 per cent for alloy No. XIV to slightly more than 20 per cent for alloy No. XXI.

CONCLUSIONS

In the case of the twelve aluminum-base alloys there was very little difference in the effect of 15 yr. of indoor or outdoor aging on the physical properties of the various alloys. Contrary to expectations, the loss in physical properties on outdoor aging did not increase in proportion to the copper content. In the case of the nine zinc-base alloys the aging characteristics and the corrosion resistance of the 3 per cent copper, 4 per cent aluminum alloys were improved by the addition of a small percentage of magnesium. Likewise, when no magnesium was added, the corrosion resistance was improved by using a higher quality of zinc in compounding the alloy. Where magnesium was added to the alloy, a further improvement was obtained by using a higher quality of zinc.

It must be borne in mind that, of the original twelve aluminum-base alloys studied, only the low-silicon alloy (No. IV), the high-silicon alloy (No. V) and a modification of the low-copper, low-silicon alloy (No. VII) are still used commercially. All the other alloys have been discontinued for one reason or another. However, based upon these aging studies there is no basis for choosing either alloys Nos. IV or VII in preference to the other alloys. Alloy No. V was somewhat less affected by indoor and outdoor aging than were the other two alloys.

In the case of the zinc-base alloys, none of the alloys studied is used commercially at the present time. A modification of alloy No. XXI with 0.03 to 0.08 per cent magnesium instead of the 0.1 per cent magnesium originally specified, is the only one of the nine zinc-base alloys used today by the die-casting industry. However, since it has been found that the lower copper content alloys compounded from Special High-Grade Zinc containing a minimum of 99.99 per cent zinc, such as the present Alloys Nos. XXIII and XXV now specified in A.S.T.M. Specifications for Zinc-Base Alloy Die Castings (B 86 – 43),[3] are vastly superior in their resistance to aging and corrosion, than are any of the alloys studied, even the modified No. XXI alloy has but limited usage today.

Acknowledgments:

The authors are indebted to the Bell Telephone Laboratories for permission to publish these data and particularly to J. R. Townsend, Materials Engineer of the Bell Telephone Laboratories, under whose guidance this work was carried out. We also wish to thank I. V. Williams and S. M. Arnold for reading the proof and offering helpful suggestions on the arrangement.

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[3] 1944 Book of A.S.T.M. Standards, Part I, p. 887.

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(7) E. H. Dix, Jr. and J. F. Keller, "Microscopic Analysis of Specimens of Die-Cast Alloys," Report of Subcommittee XV on Die-Cast Metals and Alloys, of A.S.T.M. Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys, Appendix III, *Ibid.*, Vol. 29, Part I, pp. 215–228 (1929).

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DISCUSSION

Mr. L. L. Wyman.[1]—I should like to ask Mr. Gohn, in connection with the aging tests, how these ratings compare with the data that are given in the report of Mr. Peirce's Subcommittee V of Committee B-6 on Die-Cast Metals and Alloys.[2]

TABLE XXIII.—MECHANICAL PROPERTY DATA FOR VARIOUS A.S.T.M. DIE-CASTING ALLOY SPECIMENS AFTER 15-YR. ATMOSPHERIC EXPOSURE TESTS.

Alloy	Producer	Tensile Strength, psi.		Elongation in 2 in., per cent	
		1946 Report of Committee B-6[a]	Gohn and Menges Paper	1946 Report of Committee B-6	Gohn and Menges Paper
Aluminum-Base Alloys					
No. I	C	29 500	22 420	4	2.2
	D	28 300	24 890	4	2.2
	G	30 700	27 290	3	1.9
	S	33 000	21 350	4	2.0
	W	32 300	28 310	4	3.5
	Average	30 760	24 850	3.8	2.4
No. IV	C	30 400	28 690	4	3.3
	D	26 100	23 380	2	2.0
	G	29 100	27 310	3	3.4
	S	28 000	27 990	3	3.0
	W	31 300	30 310	2	2.0
	Average	28 980	27 540	2.8	2.7
No. V	C	29 500	30 850	1	0.9
	D	26 300	25 600	1	1.5
	G	24 900	27 040	1	1.2
	S	35 600	28 740	2	1.7
	W	29 500	28 900	1	0.9
	Average	29 160	28 230	1.2	1.2
No. VII	C	36 700	29 070	3	2.0
	D	29 800	28 950	1	1.0
	G	30 900	26 510	1	1.4
	S	35 700	30 440	1	0.8
	W	33 400	30 410	1	1.4
	Average	33 300	29 080	1.4	1.3
No. VIII	C	31 800	28 940	7	4.0
	D	28 300	24 760	3	2.0
	G	28 600	26 780	4	2.1
	S	34 200	26 740	5	2.7
	W	28 100	27 260	4	3.3
	Average	30 200	26 900	4.6	2.8
Aluminum-Base Alloys (*Cont.*)					
No. IX	C	37 100	29 830	1	0.7
	D	27 500	23 730	1	1.2
	G	32 600	28 680	2	1.5
	S	37 100	29 050	2	1.0
	W	39 300	33 520	1	1.5
	Average	34 720	28 960	1.4	1.2
No. X	C	34 000	29 670	3	1.8
	D	22 800	18 170	1	1.5
	G	27 500	25 580	2	1.8
	S	31 500	26 460	3	1.8
	W	26 600	26 360	2	1.5
	Average	28 480	25 250	2.2	1.7
No. XI	C	34 500	30 510	3	1.5
	D	28 100	28 850	2	2.2
	G	33 000	28 290	2	2.5
	S	33 500	28 590	2	1.0
	W	30 300	28 220	1	1.5
	Average	31 880	28 890	2.0	1.7
No. XII	C	36 600	30 680	2	1.5
	D	32 500	30 000	1	1.3
	G	31 400	25 520	2	1.5
	S	37 300	35 100	2	1.3
	W	35 400	34 500	1	1.5
	Average	34 640	31 160	1.6	1.4
Zinc-Base Alloys					
No. XXI	D	44 300	34 430	1	1.0
	R	45 700	39 230	1	0.5
	S	44 100	31 670	1	0.5
	T	42 600	39 330	1	1.5
	Z	41 300	37 550	1	1.5
	Average	43 600	36 440	1	1.0

[a] The 1946 Report of Committee B-6 contains no data on the outdoor exposure tests on Alloys Nos. II, III, VI, XIII, XIV, XV, XVI, XVII, XVIII-A, XVIII-B, XIX, or XX.

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[1] Research Metallurgist, General Electric Co., Schenectady, N. Y.

[2] See p. 244.

MR. G. R. GOHN (*author's closure*).—In reply to Mr. Wyman's question, a comparison between our test data and those reported by Subcommittee V on Exposure and Corrosion Tests of Committe B-6 on Die-Cast Metals and Alloys[2] is possible only for aluminum-base alloys Nos. I, IV, V, VII, VIII, IX, X, XI, and XII and zinc-base alloy No. XXI since these are the only alloys listed in Mr. Peirce's report. A comparison between the committee's tension test data reported for specimens of these ten alloys after 15-yr. exposure on the tenth floor roof of the Bell Telephone Laboratories building in New York, N. Y., and data obtained by the authors is given in the accompanying Table XXIII. A study of these data indicates that the average loss in tensile strength was 3 to 17 per cent greater after 15 yr. of outdoor exposure for specimens subjected to the artificial rain than for those exposed to normal outdoor exposure.

THE NOTCH SENSITIVITY IN STATIC AND IMPACT LOADING OF SOME MAGNESIUM-BASE AND ALUMINUM-BASE ALLOYS[*]

BY J. P. DOAN[1] AND J. C. MCDONALD[1]

SYNOPSIS

The effect of a wide variety of stress concentrations and loading conditions on the strength, ductility, and toughness of a number of magnesium-base and aluminum-base alloys, was determined, attention being directed to the high-strength aluminum-base alloys. Although sensitivity indices of general applicability could not be derived, an attempt was made to draw general conclusions which would aid in estimating the performance of these materials under load.

Cast alloys of both base metals exhibited similar sensitivities on the whole. Of the two magnesium compositions tested, that with the lower total alloy content, in the heat-treated condition, appeared to have minimum sensitivity and maximum properties in the tests.

Among the wrought alloys, the aluminum alloy (4.5 per cent copper, 0.6 per cent manganese, and 1.5 per cent magnesium) was similar in sensitivity to the least sensitive magnesium alloy (3 per cent aluminum and 1 per cent zinc) and was tougher than any of the magnesium compositions. The magnesium-base alloy (3 per cent aluminum and 1 per cent zinc) can be recommended for the best combination of toughness and insensitivity.

PREFATORY NOTE.—The nature of the subject matter of this paper has necessitated the compilation of a large volume of data. This publication includes only a portion of these results, together with discussion and summary of the entire program. However, arrangements[2] have been made whereby anyone may obtain the complete paper, including those figures and tables not reproduced here. The original paper contained 38 figures and 13 tables which summarize or compare various data, an appendix (dealing with details such as form and properties of the materials used, preparation and number of specimens, range of variables covered, testing techniques, and the type of data obtained), and complete discussion of results. The table and figure numbers used in the complete text are retained in the following presentation.

The design of a load-bearing structure requires information from stress-strain curves in uniaxial stressing. On the basis of ultimate and yield strengths in tension and compression, as well as the elastic moduli (and possibly proportional limits), a specific structure can be designed, in theory at least.

The failure of a carefully designed structure may be due to one or more causes, the most common being: accidental overloading; actual loads greater than assumed; and stresses higher than calculated due to complications of construction and loading. Also, stress concentrations accompanying changes in section, joints, surface imperfections, and eccentric loading usually lower the strength and toughness of the structure.

In general, the notch sensitivity of a

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[*] Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

[1] The Dow Chemical Co., Midland, Mich.

[2] These data are available as the Addendum to the author's reprints.

material is its reaction to stress concentrations. In its broadest sense the term may be defined as the reaction to any complication of the stress state outside the uniaxial condition. Thus, notch sensitivity relates serviceability to known properties in so far as complications of the stress state are responsible for any discrepancy between service results and design calculations. Test data are required on strength, ductility, and toughness (energy to rupture) for a variety of stress states.

The ultimate goal of all studies of notch sensitivity, as broadly defined, is complete knowledge of (1) the stress state as a function of loading, geometry, and deformation, and (2) the dependence of deformation and rupture characteristics on the stress state. Several summaries and bibliographies dealing with the extensive work on these subjects are available. It suffices here to point out that theoretical, photoelastic, and experimental methods are available for providing good approximations to stress states in actual structures for deformation within the elastic range. Plastic deformation often introduces complications, but recent theoretical treatments have given mathematical expression of the laws of behavior of metals under combined stress and have clarified the dependence of fracture stress on stress state. However, anisotropy of the material and inhomogeneity of the stress field are two factors which appear difficult to handle by present methods, particularly in the case of rupture phenomena. Until these difficulties are solved, the designer must depend on his judgment based on accumulated experience with the notch sensitivity problem. As a help, the present work provides empirical data on some aluminum-base and magnesium-base alloys for a considerable variety of stress states.

Magnesium-base alloys have been successfully used in stressed members over a considerable period of time and particularly for castings has a service record been established. For wrought alloys, serviceability has also been demonstrated, though the experience has been more limited. Further knowledge is needed on what to expect from conditions which are difficult to evaluate, as for example: (1) the probable response to overloads, and (2) the probable response to design loads at points of stress concentration.

In this paper only static and impact loading are treated; the response of similar materials to repeated stressing (fatigue) is covered elsewhere (**1**).[3] Tests were conducted on typical magnesium-base and aluminum-base alloys, both cast and wrought. In the case of aluminum-base alloys, attention was directed to alloys of high strength with which magnesium alloys compete in stressed applications. A few comparison tests were also conducted on mild steel sheet. Comparison of the response of magnesium to that of aluminum, with its longer service record, provides some idea of what problems, if any, peculiar to magnesium may be encountered in service. In all cases, the comparisons were based on equal volumes of materials.

METHOD

Due to the large number of tests performed, it has been necessary to present in an appendix routine details such as the form and properties of the materials, the preparation and number of the specimens, the range of variables covered, the testing techniques, and the type of data obtained: Appendix 1 (see prefatory note) is presented in the form of an outline and thus also serves to organize

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1118.

TABLE A.—MAGNESIUM ALLOYS.

Alloy	A.S.T.M. Designation	Nominal Composition, per cent					Typical Physical Properties			
		Magnesium	Aluminum	Manganese	Zinc	Iron	Condition[a]	Tensile Strength, psi.	Tensile Yield Strength, psi.	Elongation in 2 in. per cent
					Castings					
H	AZ63	Remainder	6	0.2	3	...	AC	29 000	14 000	6
							HT	40 000	14 000	12
							HTA	40 000	19 000	5
							HTS	40 000	17 000	7
	AZ92	Remainder	9	0.2	2	...	AC	24 000	14 000	2
							HT	40 000	16 000	10
							HTA	40 000	23 000	2
							HTS	40 000	20 000	3
					Sheet					
M	M1	Remainder		1.5			a	33 000	15 000	17
							h	37 000	29 000	8
FS-1[b]	AZ31X	Remainder	3	0.3	1	<0.005	FS-1a	37 000	22 000	21
							FS-1h	43 000	33 000	11
J-1[b]	AZ61X	Remainder	6	0.2	1	<0.005	a	43 000	26 000	16
							h	47 000	34 000	9
					Extrusions					
M	M1	Remainder		1.5			As Ext.	34 000	20 000	9
FS-1[b]	AZ31X	Remainder	3	0.3	1	<0.005	As Ext.	38 000	26 000	14
J-1[b]	AZ61X	Remainder	6	0.2	1	<0.005	As Ext.	44 000	28 000	16
0-1[b]	AZ80X	Remainder	8.5	0.2	0.5	<0.005	HTA	52 000	36 000	9

[a] Symbols:
AC—As Cast
HT—Solution Heat Treated
HTA—Solution Heat Treated and Aged
HTS—Solution Heat Treated and Stabilized
a—Annealed
h—Hard Rolled and Stress Relieved

[b] -1 indicates high purity alloy.

TABLE B.—ALUMINUM ALLOYS.

Alloy	Nominal Composition, per cent								Typical Physical Properties			
	Aluminum	Copper	Iron	Magnesium	Manganese	Silicon	Zinc	Chromium	Condition[a]	Tensile Strength, psi.	Tensile Yield Strength, psi.	Elongation in 2 in., per cent
					Castings							
95	Remainder	4							T-6	36 000	22 000	5
35	Remainder			0.3		7			T-6	32 000	22 000	4
220	Remainder			10					T-4	45 000	25 000	14
					Sheet							
24S	Remainder	4.5		1.5	0.6				T	68 000	45 000	19
2S	Remainder			2.5				0.25	½H	37 000	29 000	10
5S[b]	Remainder	1.6		2.5	0.2		5.6	0.3	T	76 000	66 000	11
R301[b]	Remainder	4.5	<1	0.5		0.8	0.8		T	68 000	61 000	9
					Extrusions							
24S	Remainder	4.5		1.5	0.6				T	65 000	46 000	10

[a] Symbols:
T-4—Solution Heat Treated
T-6—Solution Heat Treated and Aged
T—Solution Heat Treated and Aged
½H—Half hard

[b] All material tested in these alloys was clad.

the work and show its scope. In the discussion of test results some description of the individual tests is given to assist in interpretation of the data.

Details of the test methods on which general statements can be made are:

1. Materials: Commercial alloys from regular production were used. Aluminum and magnesium alloys of the nominal compositions, states and typical physical properties shown in Tables A and B were included in the tests as well as mild steel with a tensile strength of 60,000 psi. and an elongation of 30 per cent in 2 in. Each specific test was made on fewer materials than the maximum.

2. Scope: The variety of tests carried out is shown in the following outline abbreviated from the appendix (see prefatory note).

I. Tension
  A. Concentric Loading
    1. Circumferential notches of varying radii, round specimens.
      a. Castings
        (1) Static — Tensile strength, reduction of area, energy to rupture.
        (2) Impact — Energy to rupture, reduction of area.
      b. Extrusions (Same properties measured as for castings)
        (1) Static
        (2) Impact
    2. Circumferential notch with depth and radius equal to 1/10 minimum diameter, round specimen, static ultimate strength.
      a. Castings
      b. Extrusions
    3. Circumferential V notch of 0.002-in. radius, round specimens, 0-1HTA extrusions; static ultimate strength.
    4. Holes in sheet; static ultimate strength and energy to rupture.
  B. Eccentric Loading
    1. Edge notches of varying depths and radii, flat specimens, static only.
      a. Castings — ultimate strength
      b. Sheet—ultimate strength
      c. Extrusions — ultimate strength and energy to rupture.
    2. Surface notch, sheet, static ultimate strength.
II. Bending
  A. Simple beam, plain and V-notched Charpy type specimens, energy to rupture.
    a. Castings
      (1) Static
      (2) Impact
    b. Extrusions
      (1) Static
      (2) Impact
  B. Cantilever beam, static ultimate torque, bend angle, energy to rupture.
    1. Symmetrical edge notches of varying radii, flat specimens.
      a. Sheet
      b. Extrusions
    2. Surface notch, sheet.
    3. Simultaneous edge and surface notches, sheet.
III. Special Tests, sheet and cast plates.
  A. Center loading of circumferentially gripped specimens, static strength, deflection, energy to rupture; impact deflection and energy to rupture.
  B. Tear Test.
    1. Static load to propagate tear.
    2. Bending tear test; impact energy to rupture.
    3. Tension tear test; impact energy to rupture.

The above order and heading designation will be followed in the discussion of results. Appendix references are given in parenthesis following the table or figure number.

3. Preparation of notches: All notches

were finished with several fine machine cuts using high-speed steel tools maintained sharp and of the desired contour by repeated inspection and hand honing. Appendix 2 (see prefatory note) deals with the effect of machining.

4. Measurement of cross-sections: Dimensions entering into calculation of the results were either by micrometer or microprojector, both of which had least scale divisions of 0.001 in. and verniers for estimating to ±0.0001 in. In some cases it was necessary to use a special chisel-shaped anvil on the micrometer.

5. Speed of testing: No precise data are available. In static tests headspeed was adjusted to yield about the same rate of load application as in ordinary tension tests. Impact tests were all on a Riehlé combination impact machine on which the striking speed was 12.8 ft. per second or 18.1 ft. per second depending on the scale used. A further complication is the fact that average rate of straining, for a given speed, depends strongly on the specimen size and shape and on the energy absorbed.

6. Calculation: In all cases in which calculation of the results involves the cross-sectional area of the test specimen, the original minimum section dimensions were used, that is, for notched bars, the area under the notch.

7. Notch effect: To aid in making comparisons between materials, the notch effect is usually expressed as a dimensionless index, called the notch factor—the ratio of the strength, ductility, or toughness in the notched state to the value of the same property determined on a similar unnotched specimen. When this method could not be used, the notch factor was obtained by arbitrary use of the simple tensile value as the base of comparison, even though the "notched" specimen was quite different in form. Thus a low notch factor corresponds to a high notch sensitivity, and *vice versa*. Orders of merit are always stated in descending order, that is, the most desirable material heads the list.

8. Testing direction: Wrought materials were tested longitudinally (with the grain) unless otherwise mentioned.

9. Specific gravity: All tests and comparisons are on the basis of equal volumes of materials, that is, no allowance is made for the lower density of the magnesium alloys.

RESULTS

(In the interests of brevity this section covers only concentric tension tests, Items IA, 1, 2, 3, and 4 of outline. Complete text is available as explained in prefatory note.)

Tension Tests—Concentric Loading of Round Specimens with Circumferential Notches of Varying Radii (IA, 1, 2, and 3):

The general shape of the specimen is indicated in Fig. A1(*a*). Notch depth (in terms of percentage of cross-sectional area removed) is constant at 43.6 per cent for castings and at 64.1 per cent for extrusions; notch radius was varied from 200 to 0.075 in. in both cases. Results are presented graphically in Figs. 1 to 5 for castings and in Figs. 6 to 10 for extrusions. Also plotted in Figs. 1 and 6 are the results of some tests (IA 2 and 3 in the appendix) using notch radii of 0.0375 in. ($\frac{1}{10}$ minimum diameter) and one test with notch radius of 0.002 in.; notch depths are 30.6 and 36.0 per cent, respectively. The materials were from different batches than used for the above-described tests. These discrepancies are indicated by the broken-line curves to the data in question.

The tensile strength *versus* notch radius curves in Fig. 1 indicate general similarity in the notch behavior of the cast alloys. Maximum notch factor occurs at greater notch radii for mag-

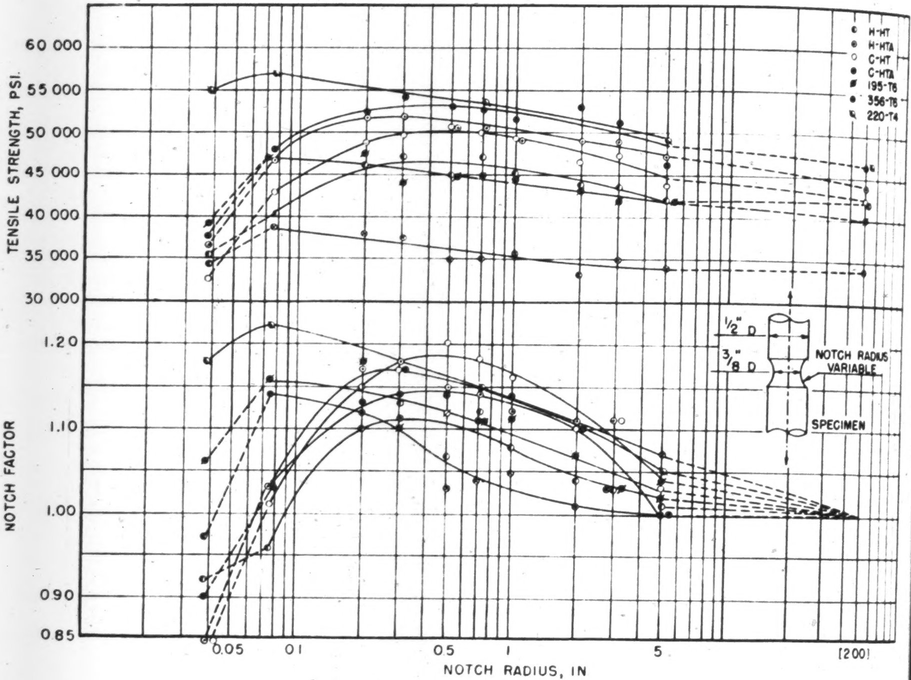

FIG. 1 (IA1a + 2a).—Static Tensile Strength *versus* Notch Radius for Castings.
Concentric loading, circumferential notches.

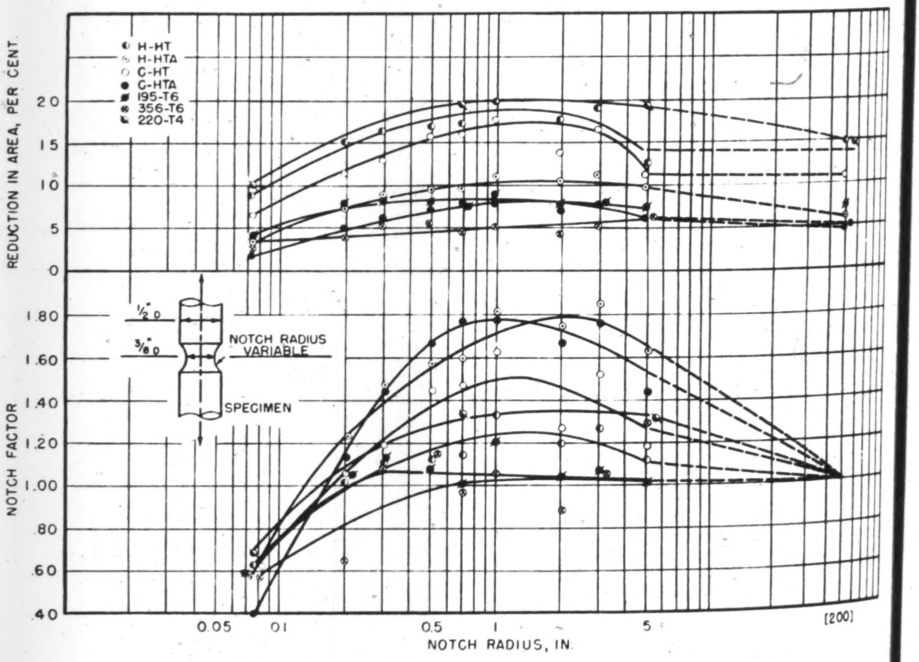

FIG. 2 (IA1a).—Static Reduction of area *versus* Notch Radius for Castings.
Concentric loading, circumferential notches.

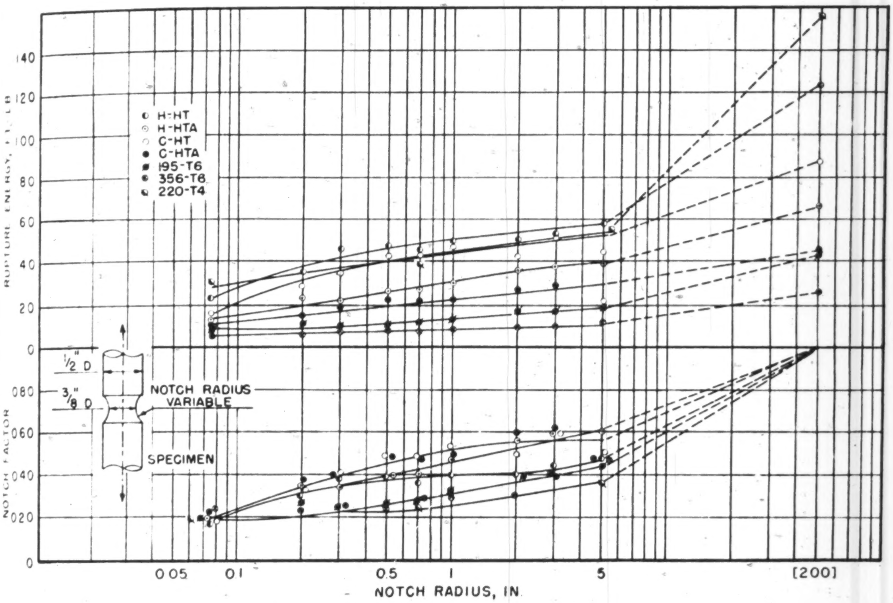

FIG. 3 (IA1a).—Static Toughness *versus* Notch Radius for Castings.
Concentric loading, circumferential notches.

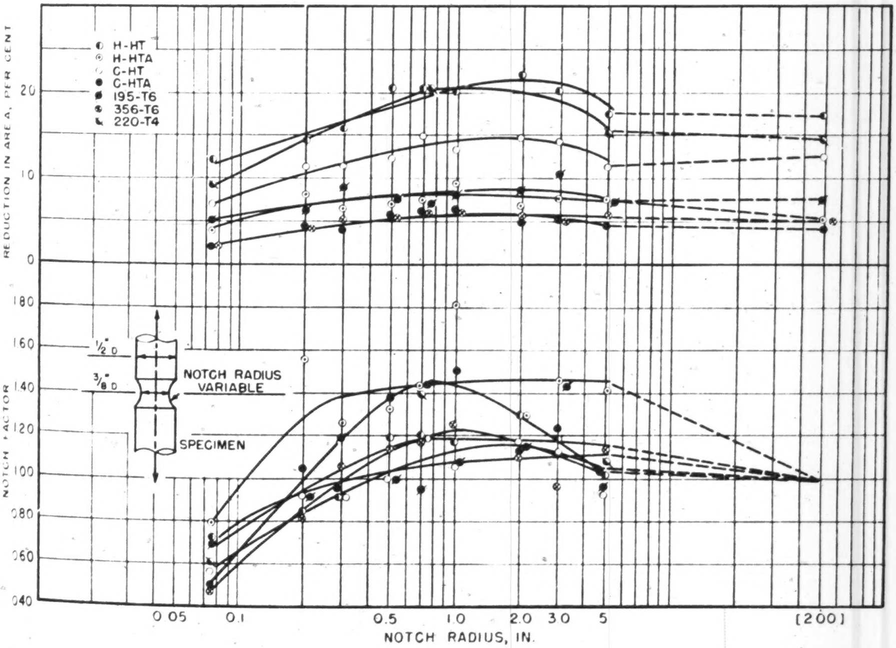

FIG. 4 (IA1a).—Impact Reduction of Area *versus* Notch Radius for Castings.
Concentric loading, circumferential notches.

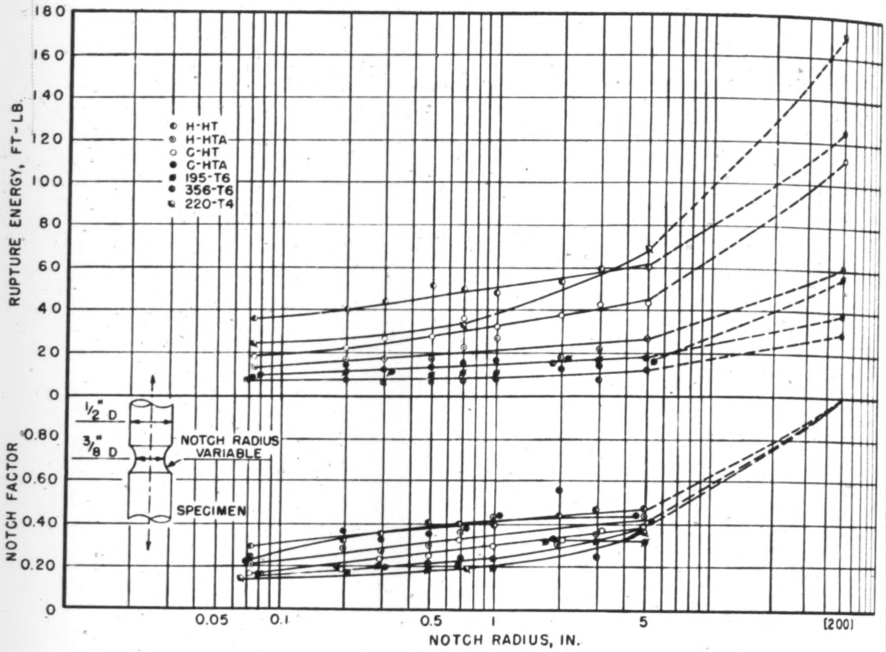

FIG. 5 (IA1a).—Impact Toughness *versus* Notch Radius for Castings.
Concentric loading, circumferential notches.

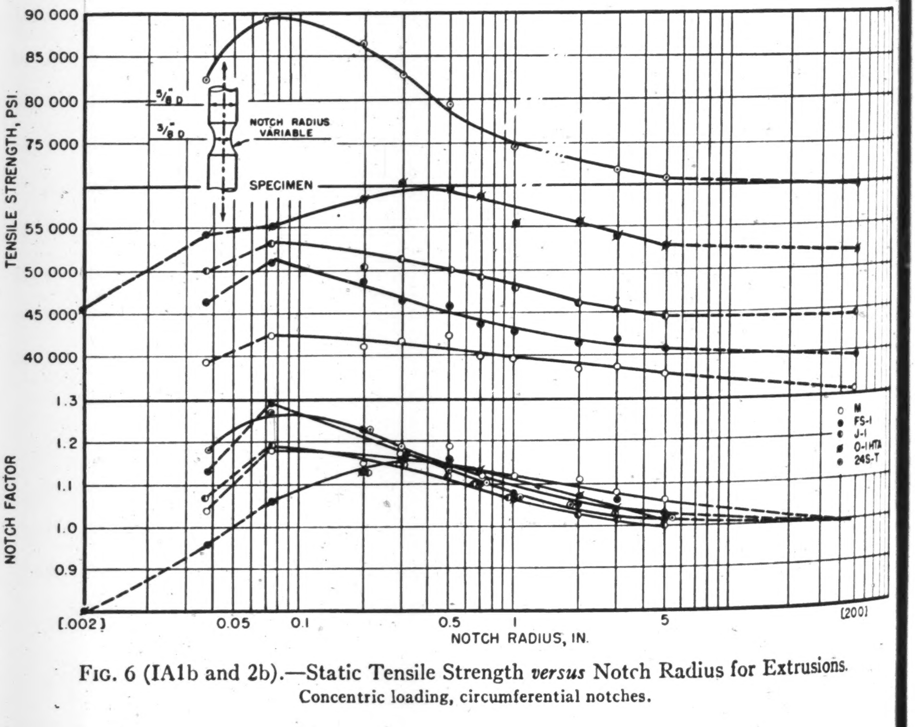

FIG. 6 (IA1b and 2b).—Static Tensile Strength *versus* Notch Radius for Extrusions.
Concentric loading, circumferential notches.

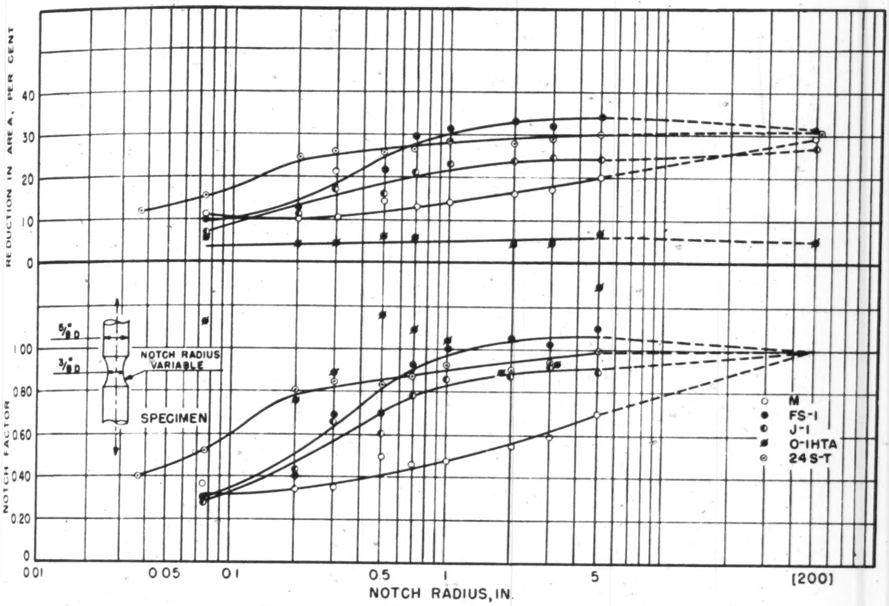

Fig. 7 (IA1b).—Static Reduction of Area *versus* Notch Radius for Extrusions.

Concentric loading, circumferential notches.

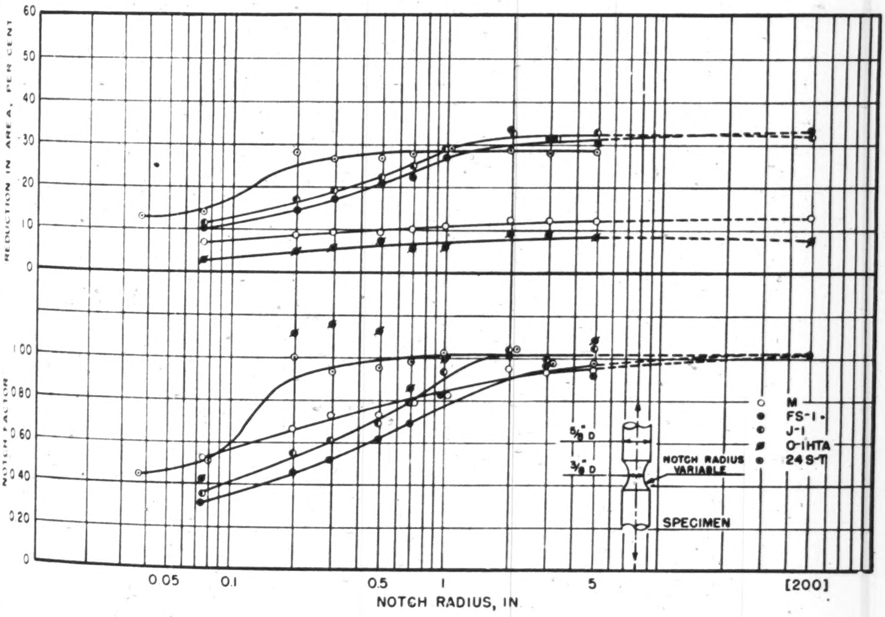

Fig. 8 (IA1b).—Impact Reduction of Area *versus* Notch Radius for Extrusions.

Concentric loading, circumferential notches.

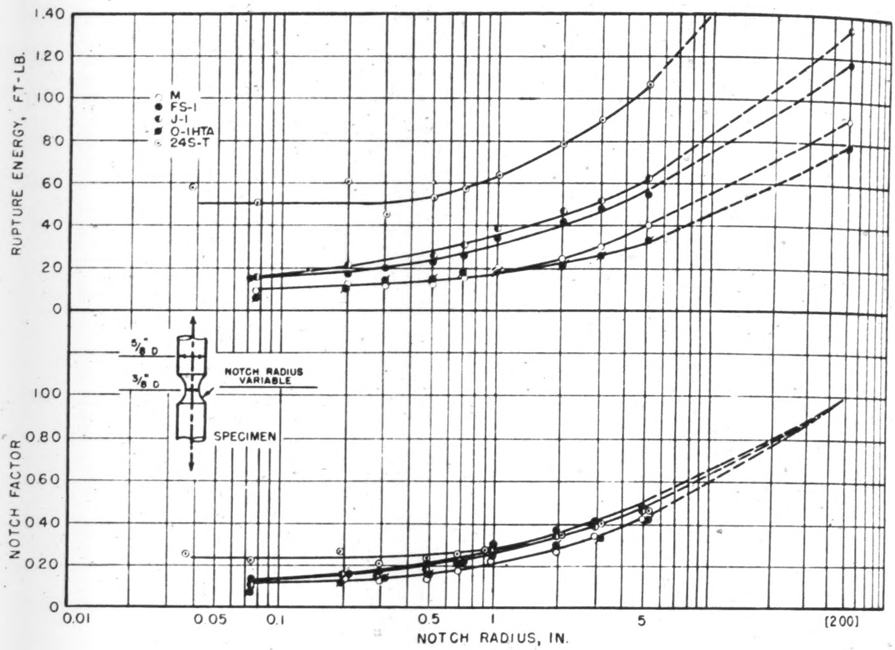

FIG. 9 (IA1b).—Impact Toughness *versus* Notch Radius for Extrusions.
Concentric loading, circumferential notches.

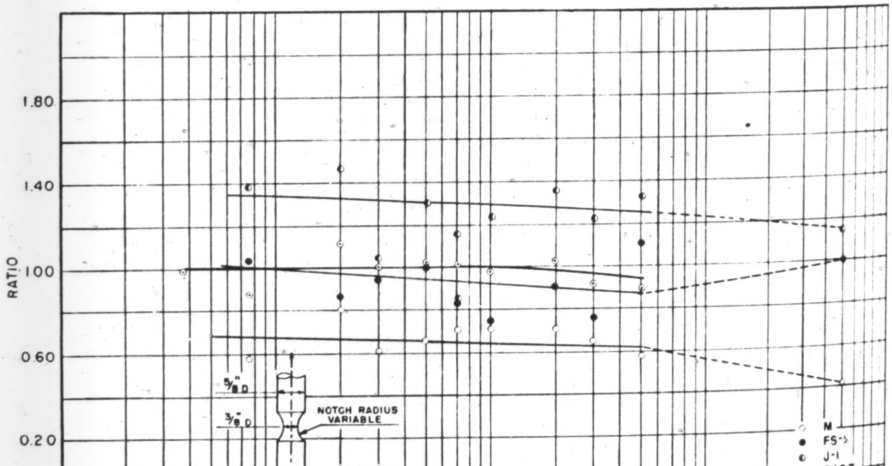

FIG. 10 (IA1b).—Ratio of Impact to Static Reduction of Area *versus* Notch.
Concentric loading, circumferential notches.

esium than for aluminum alloys. The rder of increasing average notch sensiivity is (220T-4, H-HTA, C-HT, C-HTA, 195T-6), (H-HT, 356T-6)[4] but ifferences are small.

The reduction of area curves (Fig. 2) lso show generally similar notch effects mong the alloys, particularly for the harp notches. The increase in ductility f the magnesium alloys due to mild otches is anomalous; possibly notching auses a bar to rupture at a point of verage strength whereas an unnotched ar fails at a point of weakness. Note hat the curves approach 2 to 5 per cent otch ductility at approximately the ame notch radii at which a loss in trength due to notching first occurs. This is in rough agreement with Sachs (**2**) ho shows that plastic deformation of his order is required to eliminate the nitial stress concentration. The order f increasing notch sensitivity is (H-HTA, C-HTA), (C-HT, H-HT, 220T-4), 195T-6, 356T-6). There is no clear elation between strength and ductility otch sensitivity.

Comparison of the strength and reducion of area curves with the static toughess *versus* notch radius curves (Fig. 3) eveals that the major influence of the otch on toughness is through control of he volume of metal undergoing deformation rather than through control of strength or degree of deformation. Thus, even a mild notch causes a drastic decrease in rupture energy. Alloy 220T-4 particularly illustrates this effect. Unnotched, it undergoes very uniform and extensive deformation throughout the reduced section length and thus requires a large energy rupture. Mildly notched, reduction of area is not impaired, but toughness suffers strongly due to restriction of deformation to a small volume of metal near the fracture. Here we have a case in which high tensile ductility and high notch sensitivity occur in the same material.

There are no striking fundamental differences among alloys with respect to the notch effect. Grouping the alloys in order of increasing notch sensitivity yields (C-HTA, C-HT, H-HTA), H-HT, (195T-6, 356T-6, 220T-4). Grouping in order of decreasing toughness yields (H-HT, C-HT, 220T-4), (H-HTA, C-HTA), (195T-6, 356T-6).

Tension-impact data for the cast alloys are reported in Figs. 4 and 5 showing the effect of notch radius on reduction in area and rupture energy respectively. Neither set of curves differs greatly from the corresponding static data. The impact reduction of area shows less tendency for the anomalous increase at mild notch radii. Table I gives the ratios of impact to static toughness for the cast alloys. The random scatter is consider-

TABLE I (IA1a).—RATIO OF IMPACT TO STATIC TOUGHNESS OF CAST ALLOYS.

(Concentric loading—circumferential notches)

Alloy	Condition	Ratios for Given Notch Radii										
		200 in.	5 in.	3 in.	2 in.	1 in.	0.7 in.	0.5 in.	0.3 in.	0.2 in.	0.075 in.	Average
H	HT	1.02	1.05	1.09	1.08	0.95	1.08	1.08	0.94	1.08	1.61	1.098
H	HTA	0.93	0.68	0.55	0.49	0.88	0.83	0.68	0.75	0.74	0.95	0.748
......	HT	0.77	0.99	0.82	0.88	0.76	0.84	0.65	0.75	0.76	1.12	0.834
......	HTA	0.86	0.82	0.52	0.47	0.76	0.70	0.61	0.69	0.92	0.79	0.714
95	T-6	1.32	0.96	1.09	1.13	0.78	0.97	1.15	1.07	0.85	1.05	1.037
35	T-6	1.19	1.06	0.71	0.95	0.93	0.97	1.00	0.91	1.05	1.09	0.986
22	T-4	1.15	1.23				0.90				0.78	1.015

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[4] The parentheses enclose groups of alloys having nearly al sensitivities. The actual values of the notch facs of the individual alloys and the averages for the oups are summarized in Table XIII.

able, but there appears to be a slight tendency toward relatively lower impact than static toughness, particularly for H-HTA, C-HT, and C-HTA, and for all the materials at intermediate notch radii. The orders of merit for the impact tests are:

Reduction of area notch sensitivity: H-HTA, (C-HTA, H-HT), (220T-4, 195T-6, 356T-6, C-HT).

Reduction of area: (H-HT, 220T-4), C-HT, (H-HTA, 356T-6), (C-HTA, 195T-6).

Toughness sensitivity: (H-HT, C-HTA, H-HTA), (C-HT, 220T-4, 356T-6, 195T-6).

Toughness: (H-HT, 220T-4, C-HT), (H-HTA, C-HTA, 195T-6, 356T-6).

TABLE II (IA1b).—TENSILE TOUGHNESS—STATIC VERSUS IMPACT—EXTRUSIONS.
(Concentric loading, circumferential notches)

Alloy	Notch Radius, in.	Toughness				Reduction of Area			
		Static		Impact		Static		Impact	
		ft-lb.	Notch Factor	ft-lb.	Notch Factor	Per Cent	Notch Factor	Per Cent	Notch Factor
M	0.075	9	0.11	11	0.12	5.2	0.17	4.8	0.42
	2	21	0.26	26	0.29	16.7	0.54	11.1	0.97
	200	80	....	89	....	30.7	....	11.5	....
FS-1	0.075	13	0.16	15	0.17	9.8	0.30	7.9	0.23
	2	40	0.48	38	0.43	32.1	0.99	31.5	0.92
	200	83	....	88	....	32.2	....	34.4	....
J-1	0.075	14	0.12	15	0.12	9.7	0.40	9.6	0.28
	2	48	0.41	47	0.37	31.0	1.27	33.5	0.99
	200	116	....	127	....	24.4	....	33.7	....
24ST	0.075	41	0.18	51	0.22	16.0	0.48	13.9	....
	2	77	0.34	78	0.34	28.0	0.84	28.5	....
	200	226	....	>220	....	33.5	....	....	....

Figure 6 is a plot of tensile strength *versus* notch radius for the wrought alloys. Again the curves of the various alloys are similar with minor exceptions at the sharper notches. 0-1HTA undergoes a decrease in strength at greater notch radii than the other compositions; in this respect 0-1HTA behaves much like the cast magnesium alloys. In sensitivity to the sharper notches, M and J-1 fall between 0-1HTA and 24S-T or FS-1.

The wrought alloy static reduction-of-area curves, Fig. 7, reveal that 24S-T best retains its ductility as notch sharpness increases; J-1 and FS-1 are intermediate, and M alloy loses considerable ductility due to mild notching. Similar statements hold for impact reduction of area (Fig. 8) except that M alloy has lower sensitivity due to lower ductility at 200-in. radius; it is the only alloy showing a significant reduction in ductility due to the increased strain rate of the impact test. Figure 10 clarifies this relation; contrary to the M alloy behavior, J-1 alloy actually undergoes consistently greater reduction of area in the impact test. To complete the static *versus* impact data, toughness spot checks were run on M, FS-1, J-1, and 24S-T alloys at three notch radii as reported in Table II. There is little effect of testing speed except on the reduction of area of M alloy as previously observed. Note, however, that the rupture energy of M alloy is not decreased by impact testing.

The toughness curves (Fig. 9) indicate generally similar behavior of the alloys except for the lower sensitivity of 24S-T at sharp notch radii; and over-all differences in toughness level. The rupture energy of 24S-T in this test is 2 to 5 times that of the magnesium alloys of which J-1 and FS-1 alloys are the toughest and 0-1HTA is the least tough.

Certain rough comparisons may be made with Sachs' (2) results on heat-

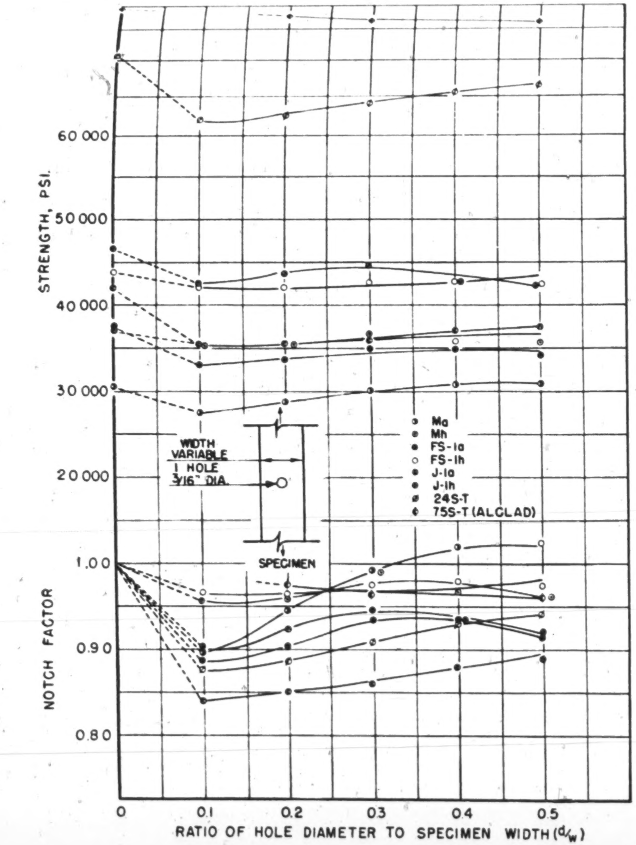

FIG. 11 (IA4).—Static Tensile Strength *versus* Hole Diameter to Specimen Width Ratio for 1-Hole Sheet.

Concentric loading.

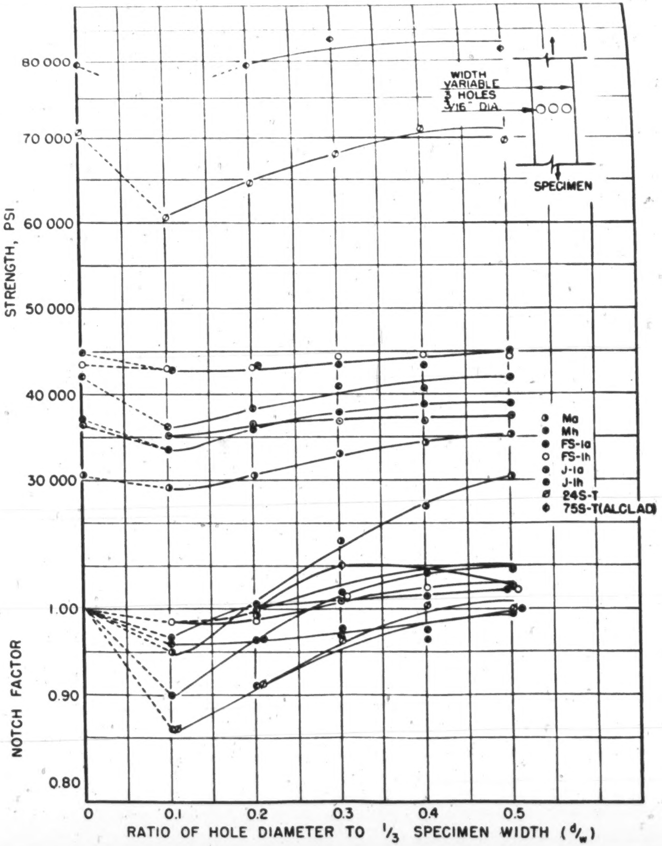

FIG. 12 (IA4).—Static Tensile Strength *versus* Hole Diameter to One Third Specimen Width Ratio for 3-Hole Sheet.

Concentric loading.

treated low-alloy steel. On S.A.E. 3140 steel heat-treated to 240,000 psi. tensile strength, he has obtained tensile strength *versus* notch radius curves of similar shape to those presented above, except that the strength increase due to notching is greater (notch factor as high as 1.5 for a 50 per cent notch) and maximum strength occurs at somewhat sharper notches than for magnesium. However, the initial rate of strength increase with decreasing notch radius appears to be approximately the same for steel, aluminum, and magnesium alloys. This substantiates Sachs' explanation of the effect as due to increasing triaxiality of the stress state, a factor substantially independent of the material, provided there is sufficient ductility to eliminate the initial stress concentration. The occurrence of the maximum strength at greater notch radii in the case of magnesium alloys is probably related to lower notch ductility and failure to eliminate completely the initial stress concentration.

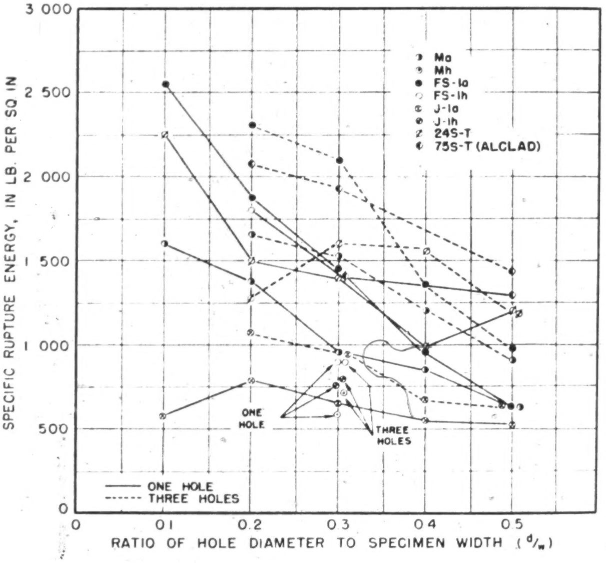

FIG. 13 (IA4).—Specific Static Toughness *versus* Ratio of Hole Diameter to Specimen Width for Sheet.

Concentric loading.

Tension Tests—Concentric Loading of Flat Specimens with Holes (IA 4):

The general shape of the specimens is indicated in Fig. A1(*b*). Hole diameter d was held constant at $\frac{3}{16}$ in. and the width of the specimens w was varied to produce the desired d/w values. In the specimens having three holes, w designates the distance between hole centers. The edge distance was equal to $w/2$. The long 12-in. reduced section was introduced as an aid in attaining axial alignment.

The relation between strength and d/w is depicted in Figs. 11 and 12 for the single-hole and three-hole tests, respectively. It should be pointed out that no attempt is made to separate the effect of width alone from that of relative hole diameter and specimen width. The unnotched specimens, on whose strengths the notch factors are based, were of standard dimensions ($\frac{1}{2}$-in. reduced section width); unnotched specimen strengths are plotted at $d/w = 0$. The discontinuity between $d/w = 0$ and

0.1 is indicated by the broken-line curves in this region. In almost every case, maximum notch sensitivity occurs in the region of $d/w = 0.1$. The less ductile alloys, Mh, FS-1h, J-1h, and 75S-T appear to have flatter curves, that is, exhibit less notch effect. Also of interest is the fact that the three-hole condition usually results in a smaller strength decrease than the one-hole condition. At $d/w = 0.3$, a common rivet hole pitch ratio, the strength of the net section is within 5 per cent of the tensile strength for all alloys, except for Ma, which exceeds this limit on the safe side. At $d/w = 0.1$, the following orders of increasing notch sensitivity prevail:

One hole: (75S-T, FS-1h, Mh), (J-1h, Ma, FS-1a, 24S-T), J-1a.

Three holes: (FS-1h, 75S-T, Mh, J-1h, Ma), FS-1a, (24S-T, J-1a). Note the superiority of M and FS-1 to J-1 alloy in both hard and soft states.

TABLE III.—TENSILE TOUGHNESS.

(a) SHEET (IA4)

Alloy	Specific Toughness, in-lb. per sq. in. × 10^{-3}			Notch Factor $d/w=0.3$	
	Unnotched[a]	$d/w=0.3$			
		1 hole	3 holes	1 hole	3 holes
Ma	10.6	0.958	1.530	0.090	0.144
Mh	4.9	0.597	0.730	0.122	0.149
FS-1a	16.1	1.440	2.090	0.089	0.130
FS-1h	10.9	0.907	0.907	0.083	0.083
J-1a	13.9	0.650	0.950	0.048	0.068
J-1h	10.6	0.749	0.770	0.071	0.073
24S-T	23.7	1.400	1.590	0.059	0.067
75S-T(Alclad)	20.2	1.410	1.930	0.070	0.096

In spite of the scatter in the rupture-energy data (Fig. 13), the following points are established: (1) for a given alloy and d/w value, the three-hole condition usually shows greater specific rupture energy than the one-hole condition; (2) the aluminum alloys fall in the same toughness range as the tougher magnesium alloys. This latter fact is particularly striking in view of the unnotched tensile toughness figures in Table III which show a considerable advantage of 24S-T over the soft magnesium alloys. Magnesium alloys must exhibit lower ductility notch sensitivity in this test. The high-strength magnesium sheet alloy FS-1h is about two-thirds as tough in this test as 24S-T. Order of increasing toughness notch sensitivity at $d/w = 0.3$ is:

One hole: Mh (Ma, FS-1a, FS-1h), (J-1h, 75S-T), (24S-T, J-1a.)

Three holes: (Mh, Ma, FS-1a), (75S-T, FS-1h), (J-1h, J-1a, 24S-T).

Again M and FS-1 are superior to J-1.

DISCUSSION

The complexity of the notch sensitivity problem was clearly evident from the test data. At present, there is no doubt of the impossibility of stating generally applicable notch sensitivity indices for any specific material. The notch effect varies with stress state in a manner that can sometimes be rationalized, but seldom, if ever, predicted. The present results, if they are to have any great utility, must be reduced to a few generalizations. This section is devoted to that task by a review of the most important results with particular attention to the practical significance of the various tests and the relative notch sensitivity of the materials tested. For this purpose notch effects on strength, ductility, and toughness will be separately discussed. The average notch factors of individual alloys in the various tests reported on are summarized in Table XIII. The trend of the discussion has not been invalidated by the lack of portions of the original test data since all of the data lead to the general type of conclusion.

Strength Notch Sensitivity:

Since all failures in the last analysis are due to strength inadequacies, strength notch sensitivity is of primary importance and will be considered in some detail.

In concentric tension tests on round notched bars, magnesium-base alloys are, in general, more notch sensitive to severe notches than aluminum-base alloys. Of the magnesium-base alloys, the cast alloys and 0-1HTA are most sensitive; FS-1 the least sensitive; and J-1 and M intermediate in behavior. However, a loss in strength occurs only under severe notch conditions unlikely to occur in service. For this reason, the results of this test are probably of minor practical importance.

The concentric tension tests on flat notched bars approximates a common service condition, for example, riveted joints. However, concentric loading is a rare thing in actual structures. The outstanding result is the fact that the hard materials are less notch sensitive than the soft materials in this test. The restriction of work strengthening is the most likely explanation for this. FS-1h is similar to 75S-T and superior to 24S-T, M, FS-1a, and J-1 alloys.

The eccentric notched tension tests simulate a common service condition. Although the actual stresses imposed are not known, the differences in alloy behavior under the same conditions is of interest. The cast magnesium-base alloys are more notch sensitive than the cast aluminum-base alloys. This result is similar to that obtained in sharply notched concentric tension tests and is undoubtedly due to the same fundamental difference, possibly a greater support effect in the aluminum alloys. Of the magnesium-base alloys, the HT states are less sensitive than the AC and HTA states. For a given state, H and C alloys behave similarly except for the AC state in which H is superior. These relations are reasonably well explained by ductility.

The same test groups the wrought alloys as follows: FS-1a sheet and FS-1

TABLE XIII.—SUMMARY OF AVERAGE NOTCH FACTORS IN THE VARIOUS TESTS.[a]

(*a*) STRENGTH

Test IA1 (Static)[a]		IA4 (1-Hole)		IA4 (3-Hole)	
Material	Notch Factor	Material	Notch Factor	Material	Notch Factor
Cast		Wrought		Wrought	
220T-4	1.14[b]	75S-T	0.99	FS-1h	0.99
H-HTA	1.13	FS-1h	0.97	75S-T	0.99
C-HT	1.12	Mh	0.96	Mh	0.97
C-HTA	1.11			J-1h	0.96
195T-6	1.10		0.973	Ma	0.95
	1.12				0.972
		J-1h	0.90		
		Ma	0.90	FS-1a	0.90
H-HT	1.07	FS-1a	0.89	24S-T	0.86
356T-6	1.05	24S-T	0.88	J-1a	0.86
	1.06		0.893		0.86
		J-1a	0.84		
Wrought					
FS-1	1.15				
24S-T	1.13				
M	1.12				
	1.133				
J-1	1.09				
0-1HTA	1.08				
	1.085				

TABLE XIII.—*Continued*

(*b*) DUCTILITY

Test IA1 (Static)		
Material	Notch Factor	
Cast		
H-HTA	1.49	
C-HTA	1.44	
		1.465
C-HT	1.26	
H-HT	1.11	
220T-4	1.11^b	
		1.11
195T-6	1.02	
356T-6	0.98	
		1.00

IA1 (Impact)		
Material	Notch Factor	
Cast		
H-HTA	1.38	
C-HTA	1.14	
H-HT	1.11	
		1.125
220T-4	1.04^b	
195T-6	1.04	
356T-6	1.01	
C-HT	0.98	
		1.014

Test IA1 (Static)		
Material	Notch Factor	
Wrought		
0-1HTA	1.02	
24S-T	0.85	
FS-1	0.81	
		0.83
J-1	0.70	
M	0.49	

IA1 (Impact)		
Material	Notch Factor	
Wrought		
24S-T	0.93	
0-1HTA	0.90	
		0.915
J-1	0.77	
M	0.74	
FS-1	0.70	
		0.737

(*c*) TOUGHNESS

Test IA1 (Static)		
Material	Notch Factor	
Cast		
C-HTA	0.46	
C-HT	0.44	
H-HTA	0.44	
		0.447
H-HT	0.37	
195T-6	0.31	
356T-6	0.31	
220T-4	0.27^b	
		0.297

IA1 (Impact)		
Material	Notch Factor	
Cast		
H-HT	0.41	
C-HTA	0.36	
H-HTA	0.33	
		0.367
C-HT	0.28	
220T-4	0.26^b	
356T-6	0.25	
195T-6	0.23	
		0.255
Wrought		
J-1	0.37	
24S-T	0.30	
FS-1	0.27	
		0.285
M	0.23	
0-1HTA	0.22	
		0.225

IA4 (1-hole)		
Material	Notch Factor	
Wrought		
Mh	0.122	
Ma	0.090	
FS-1a	0.089	
FS-1h	0.083	
		0.0872
J-1h	0.071	
75S-T	0.070	
		0.0705
24S-T	0.059	
J-1a	0.048	
		0.0535

IA4 (3-hole)		
Material	Notch Factor	
Wrought		
Mh	0.149	
Ma	0.144	
FS-1a	0.130	
		0.141
75S-T	0.096	
FS-1h	0.083	
		0.0895
J-1h	0.073	
J-1a	0.068	
24S-T	0.067	
		0.0693

[a] Tests are identified by the designation used in Appendix 1.

[b] Average of results for three notch radii only.

extrusion least sensitive, 24S-T, FS-1h sheet, Ma sheet, J-1 extrusion, M extrusion intermediate, and J-1a sheet, J-1h sheet, Mh sheet, 0-1HTA extrusion most sensitive. This result is different from that for concentric tension in that the Mg sheet alloys in the annealed state are less sensitive than corresponding alloys in the hard state. Evidently ductility is of greater importance in eccentric tension. The relatively low notch sensitivity of FS-1 alloy is again demonstrated. No fundamental difference in behavior of 24S-T and magnesium-base alloys is indicated. Tension tests of flat specimens with a sharp surface notch lead to the same conclusion. FS alloy is again less sensitive than J-1 alloy.

The torque at rupture in bending tests on flat specimens with edge notches is a measure of strength under complex stress conditions allowing considerable deformation before fracture. FS-1a, FS-1h, and 24S-T are the least sensitive, and Mh and 0-1HTA the most sensitive of the alloys tested. This is in line with the eccentric tension test results. FS-1 alloy is also the least sensitive of the sheet alloys to surface notched bend tests both with and without edge notches. 24S-T ranks close to FS-1h, slightly more sensitive in the former and less sensitive in the latter.

The punch or bulge tests, although primarily of interest from the toughness standpoint, give an indication of strength sensitivity to a biaxial stress state. Ductility strongly influences the fracture load in this test because the tensile stress for a given load decreases rapidly with increasing deflection. Thus H-HT is the least sensitive of the cast materials tested followed by H-HTS and C-HT. Differences are small among the remaining alloys.

The strength sensitivities of sheet alloys in this test show mild steel to be less sensitive to a considerable extent than 24S-T which ranks just above the least sensitive magnesium-base alloys, FS-1a, Ma, and FS-1h. FS-1 alloy again exhibits its superiority over M and J-1. Note that R301-T and 75S-T are more sensitive than 24S-T.

Strength sensitivity to tearing stresses may be of importance in determining resistance to crack propagation. Mild steel, 52S-1/2H, and 24S-T are noticeably less sensitive to tearing stresses than the magnesium alloys as a class. R301-T and 75S-T rank with the best magnesium alloys, Ma and FS-1a. Among the magnesium alloys, the annealed sheet alloys are superior to the corresponding alloys in the hard-rolled state; M and FS-1 are superior to J-1.

In summary, then, strength notch sensitivity indices of general applicability cannot be stated. Differences in behavior between magnesium-base and aluminum-base alloys are not likely to be of any great practical significance, except possibly for the most sensitive magnesium-base alloys. The cast magnesium-base alloys are slightly more notch sensitive than the cast aluminum-base alloys. The best wrought magnesium alloys compare favorably with 24S-T. Among the magnesium-base alloys the less ductile states—AC and HTA cast, h and 0-1HTA wrought—are more sensitive to eccentric notched tension. Of the strong magnesium-base sheet alloys FS-1h is consistently less notch sensitive than J-1h.[5]

Ductility Notch Sensitivity:

Ductility notch sensitivity is of interest because of its relation to formability and to strength notch sensitivity. Ductility and strength sensitivity are mutually interdependent in many cases.

Cast alloy reduction-of-area sensitivities to the sharpest notches (those causing strength decreases) in concentric tension show only minor differences. Among the wrought alloys, 24S-T behavior differs from that of the magnesium-base alloys in that ductility is maintained to lower notch radii. Very sharp notches tend to reduce 24S-T to the same sensitivity level as magnesium-base alloys.

With the single exception of 0-1HTA the wrought magnesium-base alloys have lower ductility sensitivity than 24S-T in the various bend tests on flat specimens. There are no other consistent trends in the magnesium alloy differences.

The ductility sensitivity differences

---

[5] The new magnesium-base sheet alloy, JS-1, is expected largely to replace J-1. In notch sensitivity and toughness it is intermediate between FS-1 and J-1.

among the cast alloys to the stress state in the bulge test is of doubtful significance because of the high percentage random ductility differences which may occur from batch to batch of a given alloy. Among the wrought alloys, R301-T and 52S-1/2H have outstandingly low sensitivity; 75S-T and 24S-T rank just above the best magnesium alloys, Mh and Ma; FS-1, mild steel, and J-1 alloys have the highest sensitivity.

In general, no fundamental differences in ductility notch sensitivity were noted between magnesium-base and aluminum-base alloys, with the possible exception of the wrought alloys in the bulge test.

Toughness Notch Sensitivity:

Toughness may be considered to depend on the more fundamental properties of strength and ductility. If these properties were understood completely there would be no need for studying toughness. However, it is often easier to make an energy measurement than one of strength and ductility; and, in certain practical instances, more may be known of energy input than of the resulting load and deflection. This is nearly always true for impact loading conditions; thus, toughness has come to be associated with impact. Rightly considered, rate of loading is merely one of several factors influencing strength, ductility, and their integrated product, toughness. The geometry of the specimen and loading have tremendous influence on the capacity for energy absorption and it is with these effects that we are now concerned. However, due to the general unfamiliarity with relative absolute toughness values and their apparent importance, these also deserve attention.

In concentric tension tests, toughness notch sensitivity differences among the cast alloys are small, but the aluminum alloys are slightly more sensitive than magnesium alloys. In absolute toughness, the magnesium alloys in the HT state and 220T-4 are superior, the HTA magnesium alloys intermediate, and 195T-6 and 356T-6 the least tough. The same statements hold for the impact tests. The wrought alloys show great similarity in their notch behavior except for the superiority of 24S-T under sharp notches. Absolute toughness values reveal greater differences; 24S-T is approximately two to three times as tough as the best magnesium alloys FS-1 and J-1; M and 0-1HTA are about one half as tough as FS-1 and J-1.

Concentric notched tension tests on flat sheet specimens produce relatively greater notch sensitivity in 24S-T than in 75S-T and the magnesium sheet alloys. Among the magnesium alloys, the order of increasing sensitivity is M, FS-1, and J-1. The relative sensitivities result in absolute toughness of approximately the same magnitude for FS-1a, 24S-T, and 75S-T. FS-1h, the toughest of the hard-rolled magnesium alloys, is about 60 per cent as tough as 24S-T in this test.

The alloy 24S-T is also highly notch sensitive in the eccentric notched tension test, ranking with 0-1HTA the most sensitive magnesium alloy. FS-1 is the least sensitive alloy tested; M, J-1, and 75S-T are intermediate. In absolute toughness, 24S-T and 75S-T are similar and superior to the magnesium-base alloys under mild notches; and in the same range, under sharp notches.

Charpy type toughness tests reveal similar notch behavior of aluminum and magnesium alloys in both the cast and wrought fields. The notch sensitivity differences among the alloys do not appear to be of great importance. Absolute toughness values, however, clearly show: the superiority of 220T-4 and 24S-T in the cast and wrought fields, respectively; the inferiority of C-HTA and 0-1HTA; the superiority of H over C alloy; and the superiority of the HT over the HTA state.

Bending of flat, edge notched specimens provides another case in which 24S-T is more sensitive than most of the magnesium alloys, only 0-1HTA being more sensitive. M and FS-1 are again superior to J-1. As to toughness levels among the magnesium alloys, the soft state is superior to the hard for a given alloy, and FS alloy is superior to M and J-1. 24S-T is similar to FS-1a for sharp notches and superior to it under mild notches.

In the surfaced notched bend tests, 24S-T exhibited about the same sensitivity as Mh and FS-1h. Ma and FS-1a were superior and J-1 alloy worse. Toughness values showed the usual superiority of the annealed over the hard state of magnesium alloy sheet, and of FS-1 over M and J-1. Simultaneous surface and edge notches produce greater sensitivity in 24S-T than in any of the magnesium alloys among which FS-1h and Ma are the least sensitive.

Notch sensitivity in the bulge test ranks the cast aluminum-base alloys among the magnesium-base alloys. H-HT is outstanding on the low and C-HTS on the high sensitivity side. The same holds true for actual toughness values. Among the wrought alloys R301-T, mild steel, 24S-T, and 52S-1/2H are all less sensitive than magnesium sheet alloys. Magnesium alloys are less sensitive in the soft state; and J-1 is the most sensitive of the alloys. Actual toughness values again indicate important alloy difference more clearly than sensitivity figures.

The tear toughness test results are believed to have direct bearing on the manner of failure of a complex structure. These tests measure the energy required for propagation of a crack. If this energy requirement is low, there will be a tendency for the brittle or shattering type of failure; high tear toughness, on the other hand, tends to localize the fracture.

Relative sensitivities to tearing stresses reveal 24S-T to be less sensitive than the other wrought alloys tested. Of the magnesium alloys, FS and M are less sensitive than J-1 and 0-1HTA. Cast magnesium and aluminum-base alloys fall in the same range of sensitivity, but aluminum-base alloys are consistently near the top of the range (least sensitive). As in other toughness tests, the actual toughness values may be of greater significance than the sensitivity figures. In this respect, 24S-T and H-HT are outstanding for high toughness among the wrought and cast alloys respectively. FS-1a is the toughest magnesium-base sheet alloy and FS-1h is the toughest of the hard-rolled magnesium-base sheet alloys. Mh, J-1h, 0-1HTA, and C-HTS are among the least tear tough materials tested.

Summing up the toughness tests, magnesium and aluminum-base alloys have, on the average, the same order of toughness notch sensitivity. Specific tests can be chosen which will show an advantage of either group of alloys or of an individual alloy within a group. The actual toughness values show large differences. In this consideration the cast magnesium and aluminum-base alloys fall in the same toughness range; 24S-T is considerably tougher than wrought magnesium, particularly the hard-rolled sheet alloys. Among the cast aluminum-base alloys, 220T-4 is outstandingly tough; H-HT occupies the same position among the cast magnesium-base alloys. Among the wrought magnesium-base alloy FS-1a sheet and FS-1 and J-1 extrusions are the toughest. FS-1h is the toughest of the hard-rolled magnesium-base sheet alloys.

Considering the shift in emphasis from

notch sensitivity to toughness which results from the present work, it is fitting to discuss this property in greater detail with the characteristics of magnesium in mind. The capacity for energy absorption in the elastic range is known as resilience. Toughness includes resilience plus the capacity for energy absorption in the plastic range. Both resilience and toughness may be further classified as direct or inertial as to the type of load application. Direct loads are those applied to the specimen (or structure) by an external force; inertial loads are those induced in a specimen (or structure) by the acceleration of one of its parts. Two characteristics of magnesium-base alloys give them unique advantages in toughness. The low elastic modulus enhances resilience and the low specific gravity reduces the inertial loads. As a final point, the tests and comparisons in this paper have all been on the basis of equal volumes of materials. Low specific gravity makes possible, without a weight penalty, the thickening of critical sections to lower stresses and improve their distribution.

SUMMARY

1. The effect of a wide variety of stress concentrations and loading conditions on the strength, ductility, and toughness of a number of magnesium-base and aluminum-base alloys was determined. Attention was concentrated on the higher strength compositions in the case of the aluminum-base alloys.

2. The complications of testing for notch sensitivity are such that a sensitivity index of general applicability for a given material cannot be stated. As the severity of the stress state varies, the sensitivity of a given material changes widely, with the result that the order of merit of several materials depends on the stress state.

3. Cast magnesium and aluminum-base alloys are similar in strength and ductility notch sensitivity. Aluminum-base alloys have a slight advantage in the tension type test, but H-HT is superior in the bulge test.

4. Cast magnesium and aluminum-base alloys are similar in toughness notch sensitivity. Magnesium-base alloys have a slight superiority in the tension type test and H-HT is outstanding in the bulge and tear tests.

5. As to actual toughness values, cast magnesium and aluminum-base alloys fall in the same range. The aluminum-base alloys increase in toughness relative to magnesium-base alloys as the test changes from the tension to the bending type. Alloy 220T-4 is outstandingly tough in the Charpy type test; H-HT is outstandingly tough in the bulge and tear tests.

6. As to differences among the cast magnesium-base alloys, the general superiority of H over C and of the HT state over the AC and HTA states of either alloy is indicated, particularly when toughness is desired.

7. Wrought magnesium-base alloys and aluminum 24S-T fall in the same range of strength notch sensitivity, but 24S-T is at the top of the range (least notch sensitive). Thus the best magnesium-base alloys are substantially like 24S-T; the poorest magnesium-base alloys are somewhat more notch sensitive.

8. Wrought magnesium-base alloys and aluminum 24S-T appear to be similar in ductility and toughness notch sensitivity, but these properties vary widely with stress state.

9. Wrought magnesium-base alloys are less tough than aluminum 24S-T.

10. Among the wrought magnesium-base sheet alloys, FS-1 stands out as the composition having the lowest notch sensitivity and highest toughness. In the extruded state J-1 is as good as FS-1.

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A PERMEAMETER FOR METALS USED IN CATHODE-RAY AND TELEVISION TUBES*

By Howard J. Evans[1]

Synopsis

The construction and calibration of an inexpensive permeameter for rapid routine measurements of paramagnetic alloys of low permeability are described. The permeameter can be used to measure the permeability of metal samples in the form of wire, strip, rod, tubing, or any sample whose cross-section is constant throughout its length. A typical sample of strip would be 2.5 by 0.5 by 0.030 in. Values of the quantity $(\mu - 1)$ can be determined to ± 2 per cent for metals whose permeabilities lie between 1.05 and 1.0005. Application of the permeameter is made to the measurement of the permeability of six alloys which have been used in cathode-ray and television tubes. The permeability of each alloy when fully annealed is found to be a constant independent of the magnetizing field used.

In the manufacture of cathode-ray and television tubes, it is necessary to use nonmagnetic metals. The use of any magnetic metal between the cathode and fluorescent screen might produce undesirable deflection or defocusing of the electron beam. The gun assembly, therefore, uses nonmagnetic alloys in various forms, such as wire, strip, ribbon, and tubing.

The measurement of the permeability of these materials has always presented a problem because of the inadequacy of existing test methods for materials of low permeability. The use of standard test equipment and procedures has not proved feasible, inasmuch as the raw materials as received for tube construction are generally not of the required size or shape.

This paper describes the adaptation of an absolute method (**1**)[2] to the rapid routine testing of paramagnetic materials falling within the permeability range of 1.05 to 1.0005. The permeameter described is inexpensive, easy to construct, easy to operate, and is readily adapted to the testing of metals without elaborate sample preparation.

Equipment

As may be seen in Figs. 1 and 2, the permeameter consists of an analytical balance mounted above an electromagnet. The left-hand pan of the balance is removed and a counterpoise is substituted. Directly below the counterpoise, a hole is bored in the bottom of the balance case so that a plumb line suspended from the counterpoise passes directly through the center of the air gap of the magnet. The top of the magnet housing is cut away and the space between the bottom of the analytical balance and the magnet pole pieces is surrounded by a bakelite cylinder. A section of this cylinder is cut away

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*Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

[1] Engineering Department, RCA Victor Division, Radio Corporation of America, Lancaster, Pa.

[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 1125.

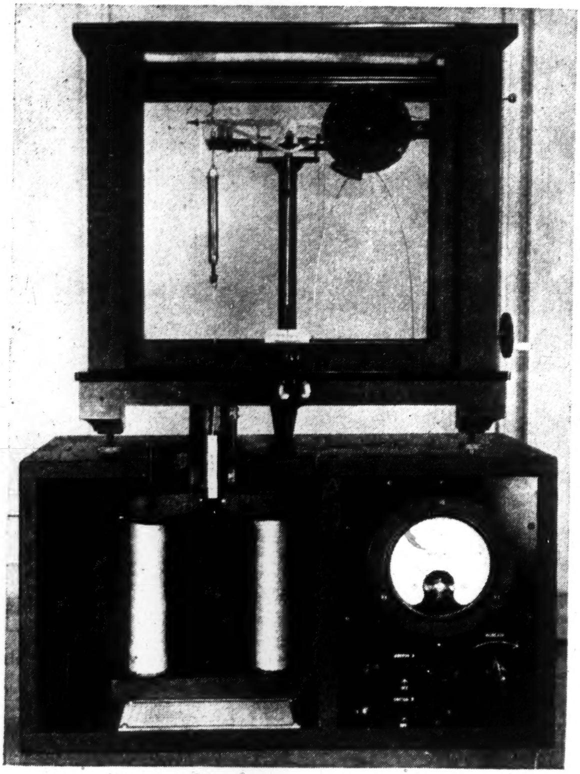

FIG. 1.—Photograph of Permeameter.

and replaced by a hinged Lucite plate which protects the sample from air currents during adjustments of the analytical balance.

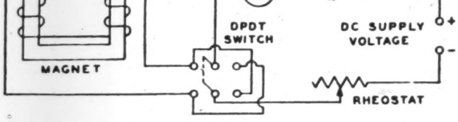

FIG. 2.—Schematic Sketch of Permeameter.

The yoke of the magnet consists of two 1-in. steel rods 8 in. long mounted on a steel base $1\frac{3}{4}$ by 1 by 8 in. The two rods are turned down to $\frac{1}{2}$ in. in diameter on each end for $\frac{1}{2}$ in. Two $\frac{1}{2}$-in. holes drilled into the base $\frac{1}{2}$ in. deep and 5 in. from center to center permit the rods to be securely fastened into the base by 8-32 machine screws threaded into the ends. Each rod has a thin brass disk press fitted on each end against the shoulder formed by the removed section. On each spool thus formed, about 1500 ft. of No. 20 General Electric Formex magnet wire is layer-wound. If smaller wire is used, the coils will heat during operation over extended periods. The yoke is capped by two pole pieces which form an air gap $\frac{3}{4}$ in. long, $1\frac{1}{2}$ in. wide, and $\frac{3}{4}$ in. deep.

The sample whose permeability is to be measured is suspended from the counterpoise between the poles of the electromagnet, and the permeability is calculated from the increase in downward force when the field is turned on. If a hole is drilled and tapped through the counterpoise, the copper wire on which the samples are supported can be fastened to a screw and the screw used to raise or lower the sample in the air gap. In this way the sample can be easily oriented so that the bottom of the sample is within $\pm\frac{1}{16}$ in. of the center line of the air gap.

THEORY OF MEASUREMENT

Gouy (2) devised a method of measuring the susceptibility of materials that are paramagnetic. In this paper, paramagnetic materials will be construed to mean materials in which there is a linear relationship between the magnetizing force and the intensity of magnetization. In the Gouy method, a sample to be tested is arranged as shown in Fig. 2. The sample is suspended from the left-hand arm of the analytical balance so that its lower end comes at the center of the air gap in a region of strong field intensity, while the upper end is in a region of much smaller field intensity. Under this arrangement it can be shown (3, 4) that the increase in force in the downward direction, F in dynes, acting on the sample is given by

$$F = 1/2\ (K_1 - K_2)\ (H_1^2 - H_2^2)\ A \ldots (1)$$

where K_1 and K_2 are the volume susceptibilities of the sample and air, respectively, in electromagnetic units, and H_1 and H_2 are the magnetizing forces in oersteds at the bottom and top of the sample respectively. A is the cross-sectional area of the sample in square centimeters. Assumptions made in the derivation of Eq. 1 require that K_1 be independent of H and that the cross-section be constant throughout the length of the sample. If the sample is sufficiently long, H_1^2 will be over 100 times H_2^2. H_2^2, therefore may be neglected in comparison with H_1^2. K_2 may also be neglected in comparison with K_1 for these measurements. Equation 1 then reduces to

$$F = 1/2KH^2 A \ldots\ldots\ldots\ldots (2)$$

where the subscripts have been dropped and it is understood that K refers to the volume susceptibility of the sample, and that H refers to the magnetizing force at the center of the air gap between the pole pieces.

In texts on electricity and magnetism (5), it is shown that

$$B = H + 4\pi I \ldots\ldots\ldots\ldots (3)$$

where B is the magnetic induction in gausses inside the material, H is the magnetizing force in oersteds, and I is the intensity of magnetization or magnetic moment per unit volume. It is customary to define the permeability μ of a material by the ratio

$$\mu = \frac{B}{H} \ldots\ldots\ldots\ldots (4)$$

If both members of Eq. 3 are divided by H, one gets

$$\mu = \frac{B}{H} = 1 + 4\pi\frac{I}{H} = 1 + 4\pi K \ldots (5)$$

where K represents $\frac{I}{H}$ and is known as the volume susceptibility. The susceptibility per unit mass is usually represented by symbol χ and is related to the volume susceptibility K by the relation $K = \rho\chi$, where ρ is the density of the magnetic material.

When all the refinements in the design of the equipment and in the technique of the measurements have been carried out, it is possible to measure K to three or four significant figures. However, in the routine testing of materials, K does not need to be known to more than two significant figures. This fact allows many of the refinements in design and in measurement technique to be neglected, as, for example, the residual magnetism of the magnetic cores when the current has been turned off.

CALIBRATION OF EQUIPMENT

The permeability can be obtained from Eqs. 5 and 2 by solving Eq. 2 for K and substituting it into Eq. 5. This gives

$$\mu = 1 + 4\pi \times \frac{2F}{AH^2} = 1 + \frac{8\pi F}{AH^2} \qquad (6)$$

A can be measured with a micrometer and F with the analytical balance so that μ can be calculated from Eq. 6 if H^2 is known.

There are two convenient ways to measure the field intensity H. One method consists of using a flux meter with a calibrated coil placed in the air gap and reading the flux density directly for any given current in the magnetizing coils. Since the steel core of the electromagnet exhibits some hysteresis (and this fact is important for measurements made with the field on), consistent measurements for a given magnetizing current can be obtained only if the field in the air gap is measured at the peak of a symmetrical hysteresis loop. To be sure that a symmetrical hysteresis loop is obtained, the reversing switch shown in Fig. 2 should be used to carry the magnet core through several cycles of its hysteresis loop for a fixed current before reading the flux meter. This procedure is essential if the same field H for a given current is always to be obtained. The values of H can be found for several different values of the current and then these same currents always used during measurements.

If a flux meter and calibrated coil are not available, the value of H can easily be found for different fixed currents by using a sample whose volume susceptibility K is known. A solution of c.p. nickel chloride is well suited for this purpose because the value of the susceptibility per gram, χ, is independent of concentration within the limits of allowable error. The susceptibility per gram of nickel chloride is given (6) by

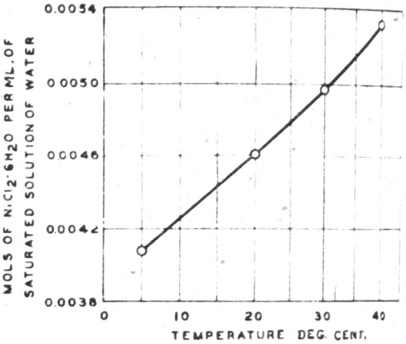

FIG. 3.—Solubility of $NiCl_2 \cdot 6H_2O$ *versus* Temperature.

$$\chi = \left[\frac{10030}{T} f - 0.720\,(1 - f)\right] \times 10^{-6} \qquad (7$$

where T is the absolute temperature of the solution and f is the fraction by weight of nickel chloride in the solution. A curve is plotted showing the concentration of nickel chloride *versus* temperature for a saturated solution. Data for such a curve were taken from the International Critical Tables (7) and are plotted in Fig. 3. From the curve, the mols per cubic centimeter of nickel chloride in solution at the desired temperature can be read directly. When multiplied by 129.6 (the gram molecular weight of nickel chloride), the weight of nickel chloride present is obtained in grams per cubic centimeter. The number of grams of nickel chloride present per cubic centimeter divided by the density of the solution gives the value of f to be used in Eq. 7. The value of K for the nickel chloride solution is obtained by multiplying the value of χ obtained from Eq. 7 by the density of the solution.

To measure H at the center of the air gap in the magnet, a small cylindrical flat-bottom glass vial is suspended from the counterpoise so that the bottom of the vial is at the center of the gap. The analytical balance is read with the field off and again with the field on. Any difference in these readings is the magnetic force on the glass. The vial is then filled with nickel chloride solution and weighed again with the field off and the field on. The difference in these two readings, when corrected for the magnetic force on the glass and converted to dynes, is the value of F used in Eq. 2. (The magnetic force on the glass vial used in the sample calculation was negligible.) The area of the inside of the cylinder may be obtained by means of a telescope gage. Since K is known for the solution, H^2 can be calculated from Eq. 2. This method of calibrating H^2 for various fixed currents in the magnetizing coils is recommended in preference to measurements made by a flux meter. It avoids the doubling of any errors in the measurement of H by the flux meter when the quantity H^2 is determined. In this, as in all measurements made with the apparatus, it is essential that the core of the magnet be carried through several cycles of its hysteresis loop for any fixed current before measurements are taken with the analytical balance.

The field in the air gap does not vanish when the current in the magnetizing coils is turned off because there is some residual magnetism in the steel cores. However, the force F which is measured with the analytical balance varies with H^2; and, therefore, any error in K caused by assuming zero field when the current is turned off will not be more than 1 per cent if the residual field is not more than one tenth of the field when the current is turned on.

Another negligible error is introduced because the field at the top of the sample does not vanish as assumed. The length of the sample necessary to make this error less than 1 per cent will depend both on the design of the magnet and the air gap. With the apparatus described herein, a sample need not be more than $2\frac{1}{2}$ in. long. Experimentally, the length can be found by taking a long sample and gradually shortening it until a difference in the attracting force of the magnet is found.

SAMPLE CALIBRATION FOR *H* *versus* MAGNETIZING CURRENT

Inside diameter of vial containing saturated solution of $NiCl_2$ at 26 C. = 0.511 in.

Cross-sectional area of vial = 1.32 sq. cm.

Current through magnetizing coils = 0.5 amp.

Weight of vial and solution with current on = 6.1454 g.

Weight of vial and solution with current off = 6.1319 g.

F, force due to magnetic field = 0.0135 g.

From Eq. 2

$$F = 1/2KH^2A$$

If K is known, then H^2 can be determined for the magnetizing current. To get K for the standard solution of $NiCl_2$, ρ and χ must be determined. The density ρ of the saturated $NiCl_2$ at 26 C. = 1.55 g. per cu. cm. From Eq. 7, χ can be obtained as follows: From Fig. 3 the number of mols of $NiCl_2 \cdot 6H_2O$ at 26 C. in 1 cu. cm. of a saturated solution is 4.81 × 10^{-3}. This number multiplied by 129.6, the gram molecular weight of $NiCl_2$, gives $4.81 \times 10^{-3} \times 129.6 = 0.623$ g. of $NiCl_2$ per cubic centimeter. The fraction by weight, f, of $NiCl_2$ present equals $0.623 \div 1.55 = 0.402$. This value for f is now substituted into Eq. 7:

$$\chi = \left[\frac{10030 \times 0.402}{273 + 26} - 0.720\,(1 - 0.402) \right] \times 10^{-6}$$

$$\chi = 13.1 \times 10^{-6}$$

$$K = \rho\chi = 1.55 \times 13.1 \times 10^{-6} = 20.3 \times 10^{-6}$$

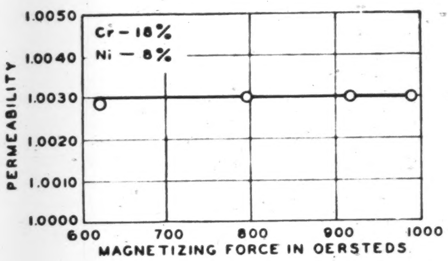

FIG. 4.—Permeability *versus* Magnetizing Force of Annealed Corrosion-Resistant Steel.

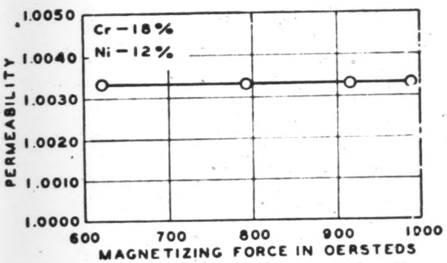

FIG. 5.—Permeability *versus* Magnetizing Force of Annealed Corrosion-Resistant Steel.

All of the values needed for Eq. 2 have now been determined. Substituting these values in Eq. 2

$$0.0135 \times 980 = 1/2 \times 20.3 \times 10^{-6} H^2 \times 1.32$$

or

$$H^2 = 98.8 \times 10^4$$

PREPARATION OF SAMPLES AND METHOD OF MEASUREMENT

The sample to be tested is cut to the desired dimensions, annealed to remove any effects of cold working, and suspended from the counterpoise. Annealing in a reducing atmosphere is necessary to eliminate scale formation. In preparing the sample, it is essential that the bottom be cut square and that the sample have a uniform cross-section throughout its length. The cross-section dimensions of the sample should not exceed one half the dimensions of the air gap.

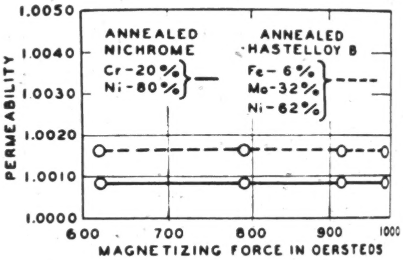

FIG. 6.—Permeability *versus* Magnetizing Force of Annealed Nichrome and Annealed Hastelloy B.

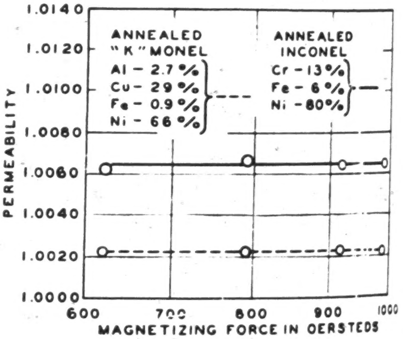

FIG. 7.—Permeability *versus* Magnetizing Force of Annealed "K" Monel and Annealed Inconel.

The sample is weighed with the current off and then again with the current set at a value chosen during the calibration. For these fixed values of current determined in the calibration, H^2 can be replaced in Eq. 6 by its numerical value. The value of μ is then given by the equation

$$\mu = 1 + \frac{CF}{A} \qquad (8)$$

where C is a constant which depends on the current used. The value of μ obtained will be independent of the current used if the material is not ferromagnetic. If the value of μ does change with current, then this method of measuring the susceptibility cannot be used. All samples should be measured with at least two different currents to see if the above condition is satisfied.

TEST RESULTS

Among the "nonmagnetic" metals which have been used for the construction of cathode-ray tube mounts are such commercially wrought alloys as corrosion-resistant steel of both the 18 chromium-8 nickel and 18 chromium-12 nickel grades, Nichrome V, Inconel, "K" Monel, and Hastelloy B. The results shown in Figs. 4 to 7 represent measurements made on samples chosen from stock. The compositions given are nominal values only and are not the result of a chemical analysis.

As has been pointed out, the results obtained with use of this permeameter will be valid only when the materials under test are paramagnetic. In order to determine whether each of the aforementioned alloys meets this stipulation, measurements were made on each sample under varying magnetizing forces. The results obtained on annealed samples when tested at room temperature (26 C.) are given in Figs. 4 to 7. It will be noted that, within the errors of measurement, the permeability of each annealed alloy remains constant over the range of magnetizing force used. Hence, these materials may be considered paramagnetic, and the values obtained with the permeameter are absolute quantitative evaluations of their permeability.

CONCLUSION

An adaptation of the highly accurate Gouy method has been found well suited for the measurement of the magnetic properties of low permeability paramagnetic alloys. It is applicable to the testing of materials of a wide variety of shapes such as those used in the construction of cathode-ray and television tubes.

The permeameter can be readily constructed in the average shop from inexpensive components, and it can be calibrated to give absolute quantitative results.

The services of a trained operator are not required to carry out the measurements, which can be made rapidly and involve a minimum of calculations.

Acknowledgment:

The author wishes to express his thanks to Mrs. H. L. Wiest for carrying out many of the measurements and to D. H. Wamsley for helpful suggestions.

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(2) L. G. Gouy, "Magnétisme sur l'énergie potentielle magnétique et la mesure des coefficients d'aimantation," *Compt. Rend.*, Vol. 9, p. 935 (1889).

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(6) H. R. Nettleton and S. Sugden, "Susceptibility of Nickel Chloride," *Proceedings of Royal Society of London*, Vol. 173, p. 313 (1939).

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CALCULATION OF ELECTRICAL CONTACTS UNDER IDEAL CONDITIONS*

By Erle I. Shobert II[1]

Synopsis

In most practical applications of electrical contacts, the choice of materials and contact forces is largely a matter of trial and error and of the experience of the designer. In this article a series of calculations is presented in which some of the variables in contacts are put in their proper relationship. As a result, the designer in certain fields of application may estimate such factors as maximum temperature rise, and knowing the mechanical and electrical characteristics of the various contact materials, he can predict the results. The calculated results are based upon contacts under ideal conditions: that is, no surface films and undisturbed smooth surfaces. The experimental data and the comparison of the predicted results with present industrial practice show that the method may be used to calculate the fundamental relations of contact resistance, contact force, and temperature rise, and that these data may be applied to practice with the proper allowance for the factors not considered in the calculations.

In a discussion sponsored by the American Institute of Electrical Engineers in Philadelphia several months ago, the author outlined some of the formulas discussed below and considered their applications to the problems of electrical contacts. H. C. Graves, Jr., of the I. T. E. Circuit Breaker Co. made some suggestions for additional calculations which were carried out and expanded. The results of this work were discussed briefly at a meeting of Subcommittee X on Contact Materials of the Society's Committee B-4 on Electrical-Heating, Electrical-Resistance, and Electric-Furnace Alloys, and the committee recommended that the work be made more comprehensive and prepared for publication. This has been done and some experimental and practical data have been provided by Fred E. Carter of Baker and Co., by H. S. Gano and H. D. Dorfman of the Westinghouse Electric Corp., by H. C. Graves, Jr., of the I. T. E. Circuit Breaker Co., by B. W. Jones of the General Electric Co., and by the Stackpole Carbon Co. These experimental data provide the practical basis for the use of the formulas and show how these formulas may be applied to the different fields of contact application.

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* Presented at a meeting of Subcommittee X of Committee B-4 on Electrical Heating, Resistance, and Related Alloys. Accepted for publication in *Proceedings*.

[1] Vice-Chairman, A.S.T.M. Subcommittee X on Contact Materials, of Committee B-4 on Electrical-Heating, Electrical-Resistance and Electric-Furnace Alloys; Research Engineer, Stackpole Carbon Co., St. Marys, Pa.

As a scientific paper, this work and all the work which has been published up to this time on electrical contacts must be considered critically. The phenomena involved in the operation and testing of electrical contacts are so complicated that there have been few examples of experimental work where all of the variables have been under control. And in these, certain variables have been eliminated which are important from a practical standpoint. Thus there is little theoretical work which has been put in a form which makes it applicable to practical design problems. On the other hand, the practical tests show such wide variations between tests and such a wide spread in the results on individual tests that it is difficult to find out what are the basic physical facts in the case.

The purpose of this paper is to describe some of the basic principles, to bring them together to form a theory of the contact resistance and its variation under different conditions, and to point a way to the application of this theory. Some experimental work is included to prove the results and to point out the difference between theory and practice.

THEORY

The problems involved in the calculations of contacts for various applications are usually carried out by rule-of-thumb methods. This is due partly to the fact that complete calculations are not available and partly because some of the factors involved in the application of contacts cannot be reduced to formulas. It is possible, however, as will be shown below, to calculate the requirements of contacts under ideal conditions. Such a solution to a contact problem must be tempered or adjusted to the practical situation by introducing safety factors in the final result to counteract the effects which are not included in the calculations. The use of these safety factors will be a matter of judgment on the part of the interested engineer or designer and will depend upon the particular field of activity. The use of the calculations which follow will be to guide the designer in the proper direction, but to leave to him the choice of safety factors to use, based on his experience in his own field of application.

This discussion is concerned with pure materials and some of the refractory base materials, with clean, undisturbed surfaces which take their characteristics only from the materials themselves and not from the surrounding conditions. Oxide layers, moisture layers, and other disturbing surface conditions are not considered in the calculations. The effect of arcing on the contact surfaces has been neglected. For some fields of application, the neglect of these factors may be compensated for by the choice of the proper safety factors, but in others further practical consideration will be required. The calculations will then indicate the magnitude of the effects which can be traced to the surface films, to the arc, and to other surrounding conditions neglected in the calculations.

For the forces usually encountered in contact work, it has been demonstrated that the force, F, in pounds, acting between two surfaces in contact is

$$F = AY \qquad (1)$$

where A is the area of contact in square inches and Y will be called the contact yield point under compression, in pounds per square inch. This formula assumes the plastic flow of contact material in the contact surface. Elastic deformation plays a part for very low contact forces, but surface irregularities make the contact even under light forces approach the case of plastic deformation.

The contact yield point as defined here is somewhat different from the usual mechanical yield point under compression. In the usual method of measuring yield point, the entire cross-section of the specimen is under uniform pressure, but in this case, since only a relatively small part of the surface area is under pressure, the material will also tend to flow laterally and the material on the sides will add to the force required to compress the small area. This definition is similar to the definitions of the Meyer hardness scale (**1**).[2]

It is believed for the purposes of the work on contacts that this concept of yield point should be adopted. A method of measuring the contact yield point may be derived directly from one of the hardness testing machines. The Rockwell Superficial Hardness Tester, for example, is being considered by Subcommittee X of Committee B-4 as the standard test for hardness of contacts.

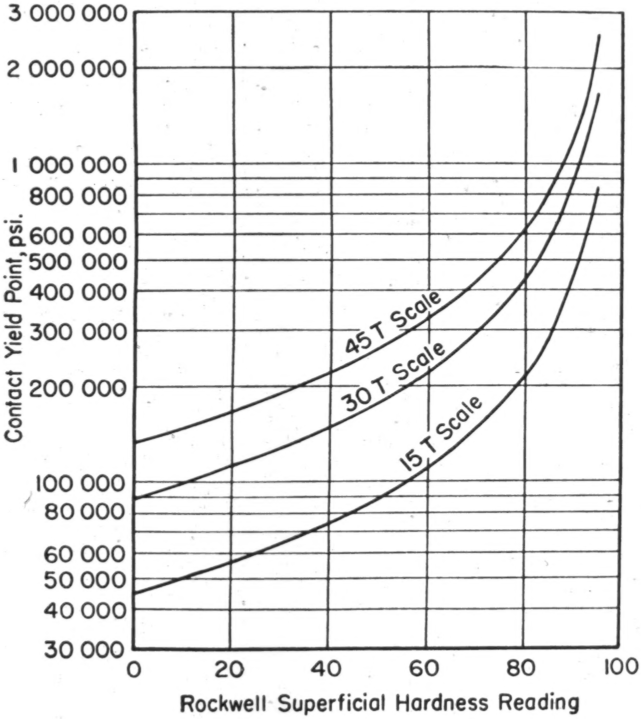

FIG. 1.—Comparison of Rockwell Superficial Hardness Scales and Contact Yield Point

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1144.

Considering only the major load on this machine, we may write,

$$L = AY \ldots\ldots\ldots\ldots (2)$$

where L is the major load in kilograms, A is the projected area of contact between the ball point and the specimen, and Y is our newly defined contact yield point. But $A = \pi a^2$, where a is the radius of the contact area. Also

$$a^2 = 2bh - h^2 \ldots\ldots\ldots\ldots (3)$$

where h is the depth of penetration of the Rockwell ball of radius b.

With the Rockwell Superficial Hardness Tester we find that

$$Y = \frac{\frac{L}{0.454}}{\pi\left[\frac{100 - R}{2.54} \times 10^{-4}\right]\left[2b - \frac{100 - R}{2.54} \times 10^{-}\right]} \text{ psi} \ldots\ldots\ldots\ldots (4)$$

where R is the hardness reading and where the constants of the dial gage have been accounted for. Figure 1 shows the relation between the Rockwell Superficial readings and the contact yield point for the different hardness scales.

TABLE I.—PHYSICAL CHARACTERISTICS OF COMMON CONTACT METALS AND A FEW OF THE COMPOSITE MATERIALS.

Material	Contact Yield Point, psi.	Rockwell Hardness	Specific Resistance, ohm-in.	Heat Conductivity, watts per in. per deg. Cent.	Wiedemann-Franz Law Constant K $\rho \times \lambda = KT$ (T = 300 K.) (v.)^2 per (deg. Cent.)^2
Common Contact Metals					
Silver (hard)	68 000	15T-34	0.64×10^{-6}	10.64	2.27×10^{-8}
(soft)	46 000	15T- 0	...	...	...
Copper (hard)	175 000	15T-75	0.68	9.86	2.24
(soft)	64 000	15T-41	...	...	...
Molybdenum	430 000	30T-80	2.2	3.71	2.72
Tungsten	665 000	30T-87	2.2	4.07	2.98
Nickel (soft)	175 000	15T-75	3.1	1.49	1.54
Graphite	6 700	15T-60[a]	320	0.399	36.2
Platinum (hard)	230 000	15T-81	3.87	1.77	2.28
(annealed)	105 000	15T-58	...	...	...
Palladium (hard)	250 000	15T-83	3.94	1.71	2.25
(annealed)	90 000	15T-51	...	...	...
Iridium	860 000	15T-95	1.94	1.50	0.97
Gold (hard)	52 000	15T-12	0.95	7.50	2.38
(annealed)	49 000	15T-8	...	...	...
Sintered Contact Materials Composition by weight					
70W 30Ag	480 000	30T-82	1 6	5.0[c]	2.67[b]
62W 38Ag	430 000	30T-80	1.2	6.5	2.60
50W 50Ag	220 000	30T-60	1.0	7.6	2.52
60Mo 40Ag	240 000	30T-64	1.3	5.9	2.54
30Mo 70Ag	170 000	30T-48	0.86	8.9	2.41
95Ag 5Graphite (hard)	120 000	15T-63	1.0	5.7	2.28
95Ag 5Graphite (soft)	89 000	15T-50	1.0	5.7	2.28
93Ag 7Graphite (soft)	81 000	15T-45	1.3	4.1	1.78
60Ag 40Ni (hard)	160 000	30T-46	1.1	5.3	1.96
40Ag 60Ni (hard)	270 000	30T-68	1.4	3.9	1.80
40Ag 60Ni (soft)	107 000	30T-15	1.6	3.4	1.80

[a] Hardness reading on Graphite taken with ½-in. diameter ball.

[b] The Wiedemann-Franz constant for the composite contact materials has been taken as the average on a volume basis of the values for the separate materials in the composition. This approximation has been made to get values to be used to calculate the heat conductivities of the composite materials in the absence of any experimental data on these heat conductivities.

[c] The heat conductivities of the composite contact materials have been calculated from the values of the Wiedemann-Franz law constant and the measured values of the specific resistance.

NOTE.—F. E. Carter, Baker and Co., supplied the data on the platinum metals and gold.

A more detailed analysis of the operation of the Rockwell hardness machines throws in question the measurement of the contact area by this method. In taking a hardness reading, the depth of penetration measurement is made with only the minor load on the ball. It might be argued that the correct depth of penetration measurement for this purpose should be made with the major load on the machine. It is believed that the standard test method is satisfactory in this case, however, since the full movement of the ball depends somewhat upon the elastic constants of the material and, to a certain extent, upon the elasticity of the machine. The measurement with the minor load should thus give, with reasonable accuracy, the depth of penetration into the material and thus the contact surface area.

TABLE II.—VALUES OF R, F, AND E WHEN $dR/dF = -0.00005$, $I = 100$ AMP., AND $n = 1$.

Material	Constant B	Contact Resistance, R, ohms	Contact Force, F, lb.	Voltage Drop at 100 amp., E mv.
COMMON CONTACT METALS				
Silver (hard)	1.48×10^{-4}	1.3×10^{-4}	1.3	13
(soft)	1.08	1.05	1.05	10.5
Copper (hard)	2.52	1.85	1.85	18.5
(soft)	1.52	1.3	1.3	13
Molybdenum	12.2	5.3	5.3	53
Tungsten	15.9	6.4	6.4	64
Nickel (soft)	11.5	5.1	5.1	51
Graphite	232	37.0	37.0	370
Platinum (hard)	16.4	6.4	6.4	64
(annealed)	11.1	4.9	4.9	49
Palladium (hard)	17.4	6.8	6.8	68
(annealed)	10.5	4.8	4.8	48
Iridium	15.9	6.3	6.3	63
Gold (hard)	1.92	1.5	1.5	15
(annealed)	1.86	1.5	1.5	15
SINTERED CONTACT MATERIALS Composition by weight				
70W 30Ag	9.81	4.6	4.6	46
62W 38Ag	6.96	3.6	3.6	36
50W 50Ag	4.15	2.6	2.6	26
60Mo 40Ag	5.63	3.2	3.2	32
30Mo 70Ag	3.14	2.1	2.1	21
95Ag 5Graphite (hard)	3.06	2.1	2.1	21
95Ag 5Graphite (soft)	2.64	1.9	1.9	19
93Ag 7Graphite (soft)	3.27	2.2	2.2	22
60Ag 40Ni (hard)	3.90	2.5	2.5	25
40Ag 60Ni (hard)	6.45	3.5	3.5	35
40Ag 60Ni (soft)	4.62	2.8	2.8	28

Values of the contact yield point are given for various materials in Table I. In general, the contact yield points are considerably higher than the conventional yield points.

Equation 1 has been checked experimentally by measuring the area of contact of crossed cylinders and of a sphere against a plane, and found to be reasonably accurate (2). It appears that with all but the very lightest loads that the material is deformed plastically even on well-polished surfaces. On the surfaces usually available on contacts, the deformation is the result of the compression of the small hills and valleys on one surface into the small hills and valleys of the other. The result is the microscopic plastic deformation of small areas, extending to a greater area, depending upon the amount of the total force. In the problem under consideration, however, we are using Eq. 2 which is similar to Eq. 1 to define our contact yield point, so that the use of Eq. 1 is reasonable from a practical standpoint.

Under ideal conditions, there will be no high-resistance film on the surface between the two contacts. The actual area of contact will behave as a perfectly conducting bridge. Since this area A is small (on pure silver with 3 lb. total force, A is approximately 5.0×10^{-5} sq. in.), we can assume a perfectly conducting sphere of radius a as the contacting medium. The contact resistance is then the resistance due only to the constriction of the current and not to the contact surface itself. This is where the ideal case departs from most practical applications, since oxide layers, moisture layers, and other films will give the contacting surfaces an appreciable resistance. The calculations which follow will indicate the best results which can be expected under the ideal conditions and will indicate the relationships between the variables when the effect of the film is neglected.

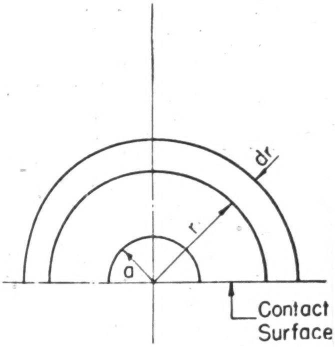

FIG. 2.—Calculation of Contact Resistance.

The resistance dR'' of the hemispherical shell between r and $r + dr$ in Fig. 2 is

$$dR'' = \rho \frac{dr}{2\pi r^2}$$

where ρ is the specific resistance of the material in ohm-inches.

The contact resistance of one side of the contact pair, R'', is

$$R'' = \frac{\rho}{2\pi} \int_a^\infty \frac{dr}{r^2} = \frac{\rho}{2\pi a}$$

or, the total contact resistance R' of both sides is

$$R' = \frac{\rho}{\pi a} \qquad \text{(5)}$$

More exact calculations (3) have been carried out without the assumption of the perfectly conducting sphere by assuming only a plane contact surface of radius a between the two surfaces. These calculations lead to the result that

$$R = \frac{\rho}{2a} \qquad \text{(6)}$$

for the total contact resistance. These equations are derived assuming that the specific resistance of the material is constant and independent of temperature. This will be seen later to be a reasonable assumption, since the temperature is limited to a small range and the specific resistance may be averaged over this range. It is also assumed that the area A is confined to one continuous region of circular section. The relations for one or more small areas whose total area is A have been worked out (4, 5). These are practical cases which probably account for the variations in contact resistance during contact tests, since the actual contact area is never the same twice.

If the contact surface area is made up of more than one contact area, there are two conditions which will exist. In the first case, the several areas will be so close together that the spreading resistance or the resistance as calculated in Eq. 6 will not be changed appreciably. In the second case, the areas will be separated to the extent that they will act independently. Assuming that the total area A is made up of n approximately equal circles of radius a_1, we have

$$A = n\pi a_1^2 = \pi a^2 \quad \ldots\ldots\ldots (7)$$

But R will be the resistance of the n small areas in parallel, or, since

$$\frac{1}{R} = \frac{n \cdot 2 \cdot a_1}{\rho}$$

then

$$R = \frac{\rho}{2a_1n} \quad \ldots\ldots\ldots\ldots (8)$$

Combining Eqs. 1, 7, and 8, we have

$$R = \frac{\rho}{2}\sqrt{\frac{\pi Y}{nF}} = \frac{B}{\sqrt{nF}} \quad \ldots\ldots (9)$$

where

$$B = \frac{\rho}{2}\sqrt{\pi Y}$$

It has been found in practice that during a test run, the contact resistance will decrease during the early period of the test. This decrease will vary somewhat from one material to another and will depend upon the application; however, in many cases, on fresh clean contact surfaces this decrease has been observed to be about 30 per cent. Other things being equal, this means that the value of n has approximately doubled. From this point on, the drop either stays low or may tend to increase as surface films are formed.

This explanation is reasonable since on freshly polished contact surfaces, particularly where a radius is placed against a plane, the contact area would be expected to be limited to a small region at the start of a test and Eq. 6 would apply, in which case n would be 1. As the contacts seat themselves in, the area within which contact could take place would be increased, causing an increase in n.

Equation 9 gives the variation of contact resistance with pressure for materials with different values of specific resistance and contact yield point. B is a constant depending only on the contact material. The validity of this equation and some of its practical applications are considered below. Values of ρ and Y are given in Table I for some of the pure metals and composite contact materials. The calculated values of B are shown in Table II.

One practical consideration which follows from Eq. 9 is a method of determining whether or not there is sufficient pressure on a set of contacts and whether or not additional pressure will be practical.

Differentiating R in Eq. 9 with respect to F, we have

$$\frac{dR}{dF} = -\frac{1}{2}\frac{B}{\sqrt{n}} \cdot F^{-3/2} \quad \ldots\ldots (10)$$

For a particular field of application a value of dR/dF may be chosen for practical purposes which will set a reasonable maximum value to the pressure for a particular contact material. Thus, for circuit breakers, a value of -0.00005 would be a reasonable limit. This means that at 100 amp. an increase in force of 1 lb. would decrease the voltage drop 5 mv. This is a reasonable practical limit for 100-amp. circuit breaker applications since the forces are about 5 lb. and the voltage drops are about 30 mv. at 100 amp. Any increase in force means added cost in the mechanism, so that the increase to be justified must bring about a corresponding improvement in the voltage drop. In other fields of application different values of these limits should be chosen.

Table II shows the values of B, R, F, and E, the contact voltage drop at 100 amp., calculated for the conditions shown above for the various contact materials, that is, for $dR/dF = -0.00005$ and $n = 1$.

It will be noticed in the analysis of the calculations in Table II that numerically the values of R, F, and E are the same. This is because of the choice of dR/dF as -0.00005, $n = 1$, and the current as 100 amp. Most other values of these quantities will eliminate this coincidence.

Temperature considerations are important in the operation of contacts and will now be considered. It has been shown that the maximum temperature rise at the junction of two contacts under ideal conditions is a function of the specific resistance of the material, the heat conductivity of the material, and the voltage E across the contacts. The general discussion of this problem may be found in the work of Slepian (**6**), Holm (**7**, **8**), and Goodwin (**9**, **10**). However, a brief derivation is given here.

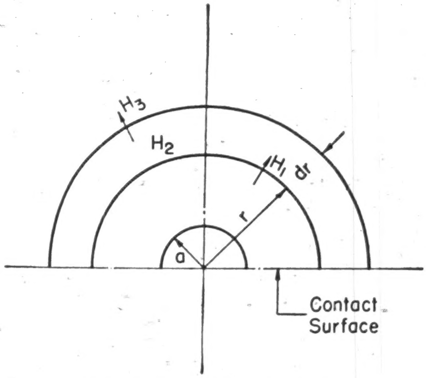

FIG. 3.—Calculation of Contact Temperatures.

The area of contact is the same as that of Fig. 2. We have the same perfectly conducting sphere of radius a, substituting for the actual contact surface. The changes in the heat conductivity and electrical resistivity with temperature are neglected since only fairly small temperature differences will be considered. H_1 in Fig. 3 is the rate of heat flow in watts passing through the surface of radius r, H_2 is the rate heat is generated in the hemispherical shell between r and $r + dr$ by the current passing through it, and H_3 is the rate of heat flow through the surface of radius $r + dr$. It is assumed that the electric current and heat flow are either parallel or antiparallel, depending upon the direction of the current. This assumption is reasonable since a is of the order of 0.007 in. ($F = 3$ lb.) for 100-amp. contacts of silver while in practice these are at least 0.090 in. thick. Thus,

$$H_3 = H_1 + H_2 \ldots\ldots\ldots (11)$$

or

$$2\pi(r + \Delta r)^2 \lambda \left(\frac{\Delta T}{\Delta r}\right)_{r + \Delta r} = 2\pi r^2 \lambda \left(\frac{\Delta T}{\Delta r}\right)_r + \frac{2\pi r^2 (\Delta V)^2}{\rho \Delta r} \ldots (12)$$

where ΔV is the voltage across the hemispherical shell of thickness Δr and λ is the heat conductivity of the material in watts per inch per degree Centigrade.

But for $r = a$, $V = E/2$, and in general

$$V = \frac{E}{2} \cdot \frac{a}{r} \ldots\ldots\ldots\ldots (13)$$

where E is the total voltage across the two contacts. Taking the limits in Eq. 12 and combining this with Eq. 13, we have

$$\lambda \frac{d}{dr}\left(r^2 \frac{dT}{dr}\right) = \frac{1}{4} \frac{E^2}{\rho} \frac{a^2}{r^2} \ldots\ldots (14)$$

One solution of this equation after supplying the boundary conditions

$T = 0$ at $r = \infty$, and $dT/dr = 0$ at $r = \infty$ and at $r = a$, is the following:

$$T = \frac{E^2}{8\rho\lambda}\cdot\frac{a^2}{r^2} \qquad (15)$$

By choosing $T = 0$ at $r = \infty$, we have made T the temperature rise of the contact above the temperature of the supporting arm back at a point where the arm is not affected by the contact. Setting $r = a$, T becomes T_1, the highest temperature in the contact junction, or

$$T_1 = \frac{1}{8}\frac{E^2}{\rho\lambda} \qquad (16)$$

The contact arm itself will have a certain temperature rise above the ambient temperature due to the current carried by the arm, and T_1 is the temperature rise of the contact surface above the conductor temperature T_c. Thus, we may write

$$T_{\text{max.}} = T_1 + T_c$$

or

$$T_{\text{max.}} = \frac{1}{8}\frac{E^2}{\rho\lambda} + T_c \qquad (17)$$

To have the physical measurements check the requirements of the theory, it would be avisable to have the conductor temperature T_c measured at a point near the contacts, and to take the voltage drop readings at the same point. From Eq. 15 it can be seen that if $a = 0.007$ in. (silver with 3-lb. force), T is 1 per cent of T_1 at $r = 0.070$. This means that the temperature difference T_1 takes place in a comparatively short distance from the contact face and that measurements of T_c and E may be made close to the contact face without introducing appreciable errors.

The assumptions which have been made in the derivation of Eq. 17 do not limit its validity and use too strenuously for contact application. The temperature rise limitations due to chemical and metallurgical considerations keep the temperature range small enough so that the equation is not too much in error because of the variations of ρ and λ with temperature. These considerations also limit the temperature to the range in which the assumption of parallel heat and current flow may be considered reasonable.

It is seen that the relation does not include any of the dimensions of the contacts. This means that regardless of the shape or size of the contacts, as long as the conditions used in the derivation are satisfied, the relation is valid. The number of small areas conducting the current across the contact surface does not affect the result if we assume that the contacting surface is all at the same potential. This assumption is reasonable on stationary contacts, but could not be made on sliding contacts. Thus, while additional contact areas will decrease the voltage drop across the contacts, the maximum temperature will still be given to a reasonable approximation by Eq. 17, which includes the contact voltage drop.

This equation by itself also gives the designer of electrical equipment a practical limit to the contact voltage drop which may be permitted on any application. It is reasonable to assume that there is a maximum temperature at the contact surface above which, due to oxidation or metallurgical change, the contacts would get into trouble. For example, on a particular circuit breaker it was noted that if the voltage drop exceeded 60 mv. across the contacts at the start of a test run, the drop would usually increase to destruction. This 60-mv. drop corresponds to a temperature rise of 60 C. in the contact surface above the terminal as calculated from Eq. 16. Add this to a terminal temperature of about 50 C. and you have the maximum temperature which may be permitted on these contacts of about 110 C.

There are several ways of setting up the limits on the temperature rise on particular contact materials. A consideration of the chemical properties including the reactions of the metals with air and the various gases which may be in the air is one method. This may be used as a check on some of the more practical methods. Another indication of a limit would be in a metallurgical change such as recrystallization which might cause the contacts to weld or stick if the surface reached this temperature.

TABLE III.—ESTIMATED MAXIMUM TEMPERATURES TO WHICH CONTACT MATERIALS MAY BE SUBJECTED

Material	Estimated Maximum Temperature, deg. Cent.	Remarks
		COMMON CONTACT METALS
Silver	200	Annealing Temperature. (Ref. "Silver in Industry," Lawrence Addicks, Reinhold, N. Y., 1940, p. 47.) This annealing or recrystallization may cause sticking. Practically this temperature is not reached because other parts in the equipment would be in trouble.
Copper	55	Copper will tarnish in air at ordinary temperatures, but this figure is taken as a practical limit based on Underwriters' Lab. and A.I.E.E. specification for bus bars which is 30 C. rise.
Molybdenum	80	Based upon maximum contact drop before failure on 15 tests on AgMo. Temperature rise calculated from Eq. 16 and added to temperature of conductors.
Tungsten	120	Based upon maximum contact drop before failure on 15 tests on AgW on relay. Temperature rise calculated from Eq. 16 and added to temperature of conductors.
Nickel	150	Based upon reaction of nickel powder with air and the increase in the reaction of H_2S and Ni at 150 C.
Graphite	225	This is the temperature at which graphite begins to oxidize slowly in air.
Platinum	400	This is the temperature at which platinum begins to recrystallize. It is probably not reached in practice because of temperature limitations of other materials in any commercial assembly.
Iridium		
Gold	150	This is the recrystallization point of pure gold. Practically all impurities tend to raise this point.
		COMPOSITE CONTACT MATERIALS Composition by weight
70W 30Ag	120	Based upon tests as listed for tungsten above.
62W 38Ag	120	Based upon tests as listed for tungsten above.
50W 50Ag	120	Based upon tests as listed for tungsten above.
60Mo 40Ag	80	Based upon tests listed for molybdenum above.
30Mo 70Ag	80	Based upon tests listed for molybdenum above.
95Ag 5Graphite	200	Based upon the component with the lower value, in this case silver.
93Ag 7Graphite	200	Based upon the component with the lower value, in this case silver.
60Ag 40Ni	150	Based upon the component with the lower value, in this case nickel
40Ag 60Ni	150	Based upon the component with the lower value, in this case nickel

A practical method of determining the values of the maximum temperature of a particular contact material is to check it on the apparatus or device in question. The current may be increased in reasonable steps and the apparatus left standing under current for fairly long periods of time. The point at which the contact voltage drop starts to increase rapidly can then be determined and the maximum temperature calculated from the ambient temperature, the temperature of the supporting parts, and Eq. 17.

The concept of the maximum temperature of a contact material must be correlated closely with the specific application. A method has been outlined above for checking this experimentally on specific applications. Some idea of the magnitudes of these temperatures might be seen from Table III in which the values are given for some materials. The source of the information is given in each case, so that the data should not be used without due consideration for its source and applicability.

If we combine Eqs. 9 and 17, we have

$$\frac{I^2}{F} = \frac{32\lambda n(T_{\text{max.}} - T_c)}{\pi \rho Y} \quad \ldots\ldots (18)$$

Fundamentally, Eq. 18 shows us that the force on contacts should increase as the square of the current for any given contact material. It also shows why it has been found desirable to use parallel contacts on very large circuit breakers, since two parallel contacts could carry the same total current with only one-half the total force. This is an important design factor since the mechanism for developing the contact forces is the major cost item. Other aspects of the importance of the factor n will be seen in the discussion of the experimental results which follow.

To get a comparison between the properties of the various materials and metals as contacts we may write

$$\frac{I^2}{F} = Dn \quad \ldots\ldots\ldots\ldots (19)$$

where

$$D = \frac{32\lambda(T_{\text{max.}} - T_c)}{\pi \rho Y} \text{ amp.}^2 \text{ per lb.}$$

Values of D for the materials listed in Table I are calculated in Table IV for which T_c is chosen 50 C. This is a reasonable choice for the conductor copper in most circuit-breaker applications.

TABLE IV.—VALUES OF THE CONSTANT D IN EQ. 19 FOR THE DIFFERENT CONTACT MATERIALS.
Conductor temperature 50 C.

Contact Materials	D, amp.^{2} per lb. of contact force
Silver (hard)	370 000
(soft)	550 000
Copper (hard)	4 200
(soft)	11 500
Molybdenum	1 200
Tungsten	1 980
Nickel (soft)	2 800
Graphite	330
Platinum (hard)	6 960
(annealed)	15 200
Iridium	......
Gold (hard)	150 000
(annealed)	164 000
70W 30Ag	4 650
62W 38Ag	9 000
50W 50Ag	24 600
60Mo 40Ag	5 800
30Mo 70Ag	18 600
95Ag 5Graphite (hard)	72 500
95Ag 5Graphite (soft)	98 000
93Ag 7Graphite (soft)	77 000
60Ag 40Ni (hard)	31 000
40Ag 60Ni (hard)	10 500
40Ag 60Ni (soft)	20 000

It will be noticed in Table IV that from the standpoint of carrying current under ideal conditions, silver is much better than any of the other materials. While the order of merit of the materials is somewhat arbitrarily based upon the choice of values for $T_{\text{max.}}$ which should be chosen differently for different fields of application, the general trend of the values is reasonable from a practical standpoint. There are several important points which can be seen from this table. These are as follows:

(*1*) The outstanding advantages of silver from a current conducting standpoint are clearly shown.

(*2*) Wide variations in the constant D are brought about by changes in the composition and hardness of the composite materials. These changes show that composition must be under close control and that processing methods will vary the results in a radical manner. These facts are also borne out by practical experience.

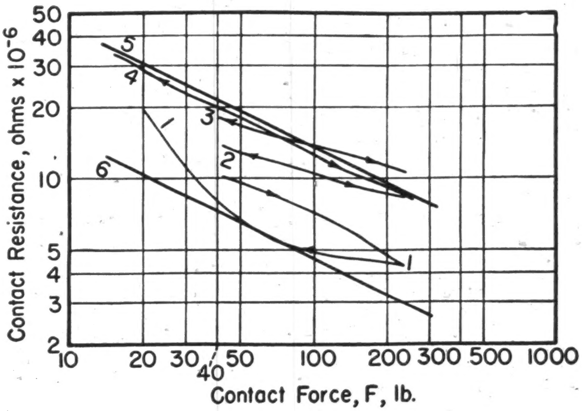

FIG. 4.—Contact Resistance *versus* Force Characteristic for Copper. (I. T. E. Circuit Breaker Co.)

1. Sand papered smooth and greased—flat.
2. Sand papered smooth and greased—line contact.
3. Ground line contact not greased.
4. Sand papered smooth and greased—edges beveled.
5. Plotted from Eq. 9, $Y/n = 50{,}000$.
6. Plotted from Eq. 9, $Y/n' = 5800$.

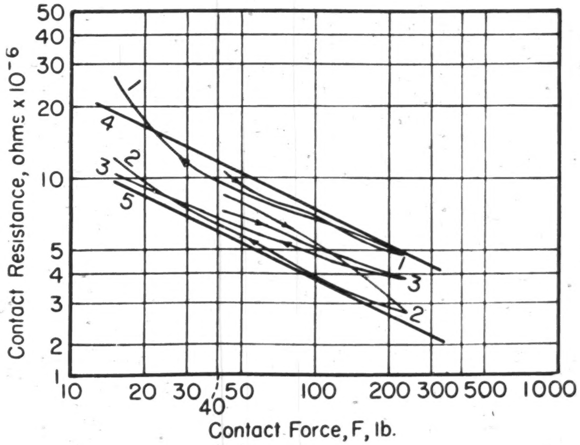

FIG. 5.—Contact Resistance *versus* Force Characteristic for Silver-Plated Copper. (I. T.E. Circuit Breaker Co.)

Stock Contacts except silver-plated $1^3/_{16}$ by $^5/_{16}$ in., carrying 500 amp. d. c.

1. Sand papered, smooth, greased, edges beveled.
2. Sand papered, smooth, greased, edges beveled.
3. Ground to make line contact, greased.
4. Plotted from Eq. 9, $Y/n = 15{,}500$.
5. Plotted from Eq. 9, $Y/n = 4000$.

(3) Silver imparts its most important advantage, that is, low drop, to the compositions it enters into.

(4) The advantages of the platinum metals and gold, since they are not influenced by atmospheric conditions, may be seen.

EXPERIMENTAL RESULTS

The first data to be considered will be that in relation to Eq. 9

$$R = \frac{\rho}{2}\sqrt{\frac{\pi Y}{nF}}$$

Figure 4 is based upon data supplied by H. C. Graves, Jr., of the I. T. E. Circuit Breaker Co., plotted on the log–log scale. The resistance force characteristic curve should be a straight line with a slope of $-\frac{1}{2}$ under these conditions.

In the first place, there is little correlation between the surface treatment and the results on these tests, although the lowest drop is experienced on flat contacts properly cleaned. Generally, the resistance force curve follows the slope predicted, at least within practical limits. It varies, however, widely in the value of the constants involved. From Table I, we see that the contact yield point for hard copper is 175,000 psi. and for soft copper 64,000 psi. This is a difference of a little over $2\frac{1}{2}$ to 1, which if placed in Eq. 9 gives a ratio of 1.6 to 1. This obviously cannot account for the values listed. Thus, the variation enters in the value of n. In **Fig.** 4 it is seen that the value of n must therefore change in the order of 10 to 1 between the various tests. This is an important point, since the value of n is largely a function of the design of the equipment and the contacts and the way they operate.

While oxides may cause some variation in the results of these tests they are not a major factor since the contact resistance values measured are all below the values to be expected for soft copper with $n = 1$, in which case Y/n would be 64,000 psi.

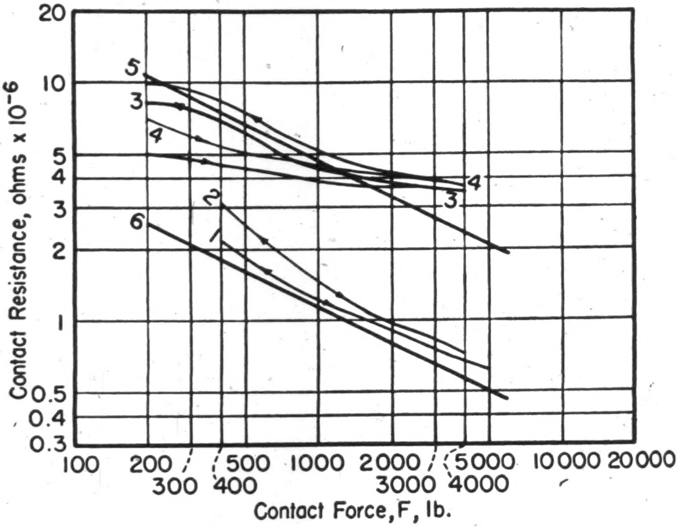

FIG. 6.—Contact Resistance *versus* Force Characteristic for 85Ag 15Ni. (I. T. E. Circuit Breaker Co.)

1. Total contact surface 13.25 sq. in., 5000 amp., d. c.
2. Total contact surface 13.25 sq. in., 5000 amp., d. c.
3. Total contact surface 2.9 sq. in., 1500 amp. d. c.
4. Total contact surface 2.9 sq. in., 1500 amp. d. c.
5. Plotted from Eq. 9, $Y/n = 44{,}800$.
6. Plotted from Eq. 9, $Y/n = 2580$.

The effect of silver plating copper contacts is shown in Fig. 5. The silver plating effectively narrows the spread in contact resistance and decreases it generally. The values in this test and those for silver contacts will be compared below. The curves for silver-plated copper are more consistent and follow the slope required by Eq. 9 closer than do the curves for plain copper shown in Fig. 4.

The same type of curve at much higher contact forces is shown in Fig. 6, for tests on an 85 per cent silver, 15 per cent nickel contact material. There seems to be some tendency for the curves to flatten out and deviate from the theoretical slope at very high forces. This might be expected since the area of contact A becomes a larger part of the area of the contact surface. This means that the infinity conditions used in the derivation of Eq. 9 are no longer valid. In any case, however, the results are close enough to the theoretical curves to be used for practical calculations as far as the variation of contact resistance with pressure is concerned.

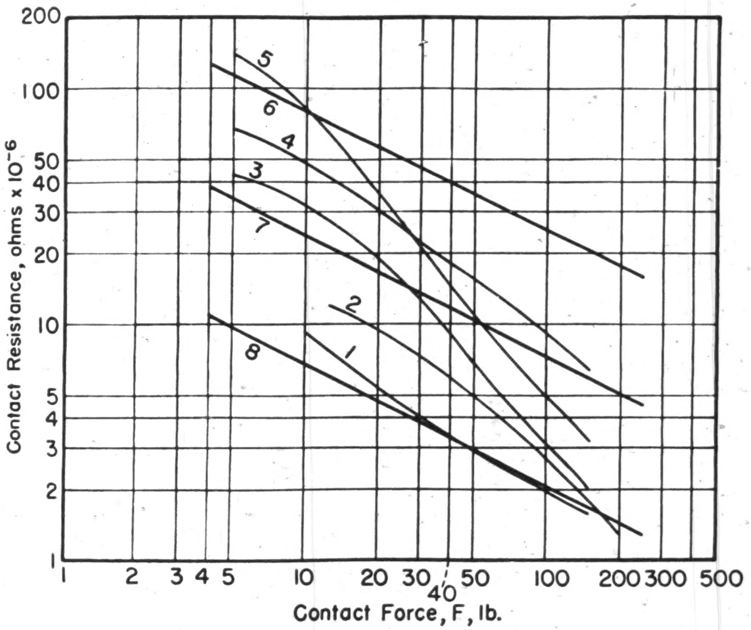

FIG. 7.—Contact Resistance *versus* Force Characteristic for Copper. (General Electric Co.)

1. Line contact 1 in. long, 3-in. cylindrical radius, dry.
2. Flat contact 3 by 3-in. surface, scraped flat on ground cast iron, dry.
3. Flat contact 1 by ¼ in., glass finish, dry.
4. Flat contact ½ by ½ in., glass finish, dry.
5. Flat contact 1 by 1 in., glass finish, dry.
6. Plotted from Eq. 9, $Y/n = 175{,}000$.
7. Plotted from Eq. 9, $Y/n = 14{,}200$.
8. Plotted from Eq. 9, $Y/n = 1180$.

Another series of experiments on copper furnished by B. W. Jones of the General Electric Co. is reported in part in Fig. 7. The purpose and the methods in this series of tests were somewhat different than those reported by I. T. E. in Fig. 4. The I. T. E. tests are probably nearer the practical surfaces available on new copper contacts. The G. E. tests were conducted to find the effects of different surface treatments. There seems to be little correlation between the results of the various tests. It will be noted from Fig. 7 that the slopes are steeper than would be expected from Eq. 9. This variation could be accounted for by assuming that on the surfaces prepared by G. E. the value of *n* was not constant during test, but increased with increased force. This explanation is reasonable since curve No. 1 in Fig. 7, which is for a line contact, follows the predicted slope quite closely. On a line contact, the value of *n* would not change as readily as on flat, glass-smooth contacts.

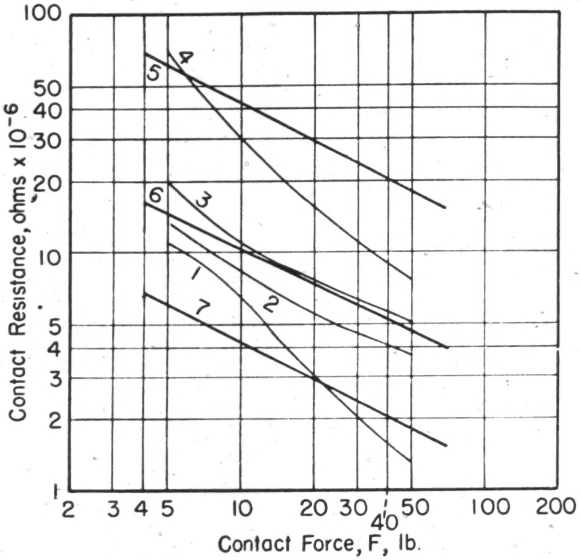

FIG. 8.—Contact Resistance *versus* Force Characteristic for Silver. (General Electric Co.)

1. Silver-faced contacts 1 by ⅜ in. flat.
2. Silver-faced 300-amp. contactor tip (soft silver).
3. Silver-faced 300-amp. contactor tip (soft silver).
4. Silver-faced 300-amp. contactor tip (hard silver).
5. Plotted from Eq. 9, $Y/n = 46{,}000$.
6. Plotted from Eq. 9, $Y/n = 3600$.
7. Plotted from Eq. 9, $Y/n = 584$.

A similar series of tests is reported by G. E. in Fig. 8 on silver contacts. It was possible to identify curve No. 4 as having been carried out on silver that was much harder than that on the other tests. As expected from Eq. 9, the resistance values are the highest. The same divergence from the slope predicted by Eq. 9 is noticed for two of the curves which is probably due to an increase in *n* with increased force.

A comparison between Figs. 5 and 8 shows that the values for silver-plated copper in Fig. 5 approximate those for the hard silver, although the slope of the copper tests is more consistent and closer to Eq. 9. This would be expected since copper is harder than silver in general. It also indicates that as long as the plating is intact, the contact resistance properties of silver-plated copper should approach those of hard silver. It also indicates that soft solid silver will always be better if *n* can be kept sufficiently large.

In the original derivation of Eq. 9 it has been assumed that the area of contact is determined by plastic flow of the contact material under specific pressures higher than the yield point of the material. With this in mind, the question is raised as to why the contact resistance increases when the force is decreased. If plastic flow alone accounted for the results, the contact area should remain unchanged and the contact resistance should remain at the lowest value as the force is decreased. The tendency in this direction is shown in Fig 4, curves 1 and 2, and Fig. 5, curves 2 and 3. On the other hand, the reverse is true in Fig. 6, curves 3 and 4, and no appreciable difference is noted in Fig. 4 curve 3, Fig. 5 curve 1, and Fig. 6 curves 1 and 2.

The reason for the increase in contact resistance as the pressure decreases may be described as follows: The actual contact surface is deformed plastically, as shown by the slope of the curves, when force is applied. This plastic deformation extends into the material for a short distance. The pressure decreases with the distance inside the material until an area is reached where elastic deformation alone can support the load. This area can be calculated from the elastic limit of the material. From this point on the deflection of the material is elastic, and Hooke's law will be obeyed. Now, if the force on the contacts is decreased, the elastic strain in the material causes the area of the limit of plastic deformation to decrease. By moving back toward the contact surface the material under elastic stress changes the area of the surface under plastic deformation and decreases the actual contact area. Thus, the two effects really work together. The plastic deformation under stress determines the area of electrical contact, and the elastic strain in the material prevents this area from being greater than that determined by the forces operating at the time of the measurement.

Some interesting results might be observed if these effects were considered in connection with work-hardening materials.

TABLE V.—CONTACT CHARACTERISTICS OF WESTINGHOUSE 100-AMP. BREAKERS COMPARED WITH CALCULATED DATA.

Breaker	Contacts		Contact Force, *F*, lb.	Minimum Measured Contact Drop, *E*, mv.	Production Maximum Limit of Contact Drop, *E*, mv.	Force and Contact Drop Calculated from Table II		Contact Drop Calculated from Eq. 9. $n = 1$ *E*, mv.
	Stationary	Movable				*F*, lb.	*E*, mv.	
No. 1..	62W 38Ag	62W 38Ag	2.75	20	80	3.6	36	40
No. 2..	93Ag 7C	40Ag 60Ni (Hard)	5	17	60	2.9[a]	29[a]	20[a]
No. 3..	95Ag 5C (Hard)	62W 38Ag	4	19	50	2.9[a]	29[a]	25[a]

[a] Average of Force and Contact Drop for the two materials in Table II.

It is also possible that the consideration of contact areas and their dependence upon the various factors *Y*, *n*, and *F*, might lead to some interesting results in work on friction.

Some contact-drop data on 100-amp. circuit breakers have been supplied by H. D. Dorfman of the Westinghouse Electric Corp. Table V shows a tabulation of the characteristics of three different types of breakers all rated at 100 amp.

In the first place, it is seen that the contact force and contact drops calculated in Table II are of the proper order of magnitude. It is also seen that values of contact drop calculated from the physical properties of the materials given in Table I and the contact forces used on these breakers also are of the proper order of magnitude. It appears that values of n in the range from 1 to 4 will account for the minimum measured contact drop and that safety factors of from 2 to 3 will account for the difference between the calculated drop and the production maximum limits. With this order of agreement, it is believed that the calculations may be used to predict the fundamental method of variation of the contact characteristics.

Our next problem is to see how closely the results calculated from Eqs. 16 and 17 on contact temperature fit the practical cases. H. S. Gano of the Westinghouse Electric Corp. has supplied some data on small circuit breakers which are reported in Table VI. Three different types of small breakers are listed with their characteristics including contact materials, contact forces, contact drop, and temperature-rise tests. The temperatures were measured as near the contacts as was practical by inserting thermocouples into small holes next to the contacts. The calculated values of $T_{max.}$ are figured by adding the temperature rise calculated from the contact drop and Eq. 16 to the measured temperatures of the arms. Where the temperatures of the two arms varied widely, an average has been taken, weighted toward the higher value.

TABLE VI.—CONTACT CHARACTERISTICS OF WESTINGHOUSE CIRCUIT BREAKERS.

Breaker	Rating	Contacts		Contact Force, F, lb.	Contact Drop, E, mv.	Measured Temperature Near Contacts, deg. Cent.		Calculated $T_{max.}$, deg. Cent.	$T_{max.}$ Table III, deg. Cent.
		Stationary	Movable			Stationary	Movable		
No. 1..	25A	70W 30Ag	70W 30Ag	0.75	30	25	42	50	120
No. 2..	50A	62W 38Ag	70W 30Ag	1.13	50	32	48	90	120
No. 3..	50A	60Mo 40Ag	60Mo 40Ag	3.0	40	25	38	58	80

It is seen from a comparison of the calculated values of $T_{max.}$ and those from Table III that these breakers are all rated safely as far as temperature rise is concerned, based upon the assumptions which have been made in the theory and calculations. Since the value of the temperature rise in the contacts as calculated from Eq. 16 varies with the square of the contact drop, a simple calculation will show what the maximum drop could be to keep the value of $T_{max.}$ below the limits for the material.

Another set of data on the same subject has been provided by H. D. Dorfman of the Westinghouse Electric Corp. on three different types of circuit breakers all rated at 100 amp. These data are on the same breakers and are the same in part as those in Table V and are shown in Table VII. On these breakers also it is seen that the calculated values of $T_{max.}$ are well below the theoretical limit. In this case, however, the contact supporting arms are at higher temperatures, and since the contact drops are lower, the temperature increases due to the contact drop are lower. The amount that the contact drop may increase without causing difficulty is also considerably lower.

A comparison of the contact resistance under operating conditions on various contact materials is

TABLE VII.—CONTACT CHARACTERISTICS OF WESTINGHOUSE CIRCUIT BREAKERS.

Breaker	Rating	Contacts		Contact Force, *F*, lb.	Contact Drop, *E*, mv.	Measured Temperature Near Contacts, deg. Cent.		Calculated T_{max}, deg. Cent.	$T_{max.}$ Table III, deg. Cent.
		Stationary	Movable			Stationary	Movable		
No. 1...	100 A.	62W 38Ag	62W 38Ag	2.75	20	74	83	90	120
No. 2...	100 A.	93Ag 7C	40Ag 60Ni (hard)	5	17	54	54	61	120
No. 3...	100 A.	95Ag 5C	62W 38Ag	4	19	62	62	69	120

TABLE VIII.—CONTACT RESISTANCE DURING LIFE ON GRAVITY-TYPE RELAY.

Contacts operating on 120 v. a. c. Contact Force 0.44 lb.

Material	Average Contact Resistance on Test, ohms × 10^{-3}	Contact Resistance Calculated Eq. 9 ($n = 1$) ohms × 10^{-3}	Type of Load
Silver (hard) . .	0.30	0.22	8000 w. lamp bank
70W 30Ag.....	1.40	1.49	8000 w. lamp bank
62W 38Ag.....	0.78	1.05	8000 w. lamp bank
60Mo 40Ag....	0.63	0.85	8000 w. lamp bank
95Ag 5Graphite (hard)	0.29	0.46	8000 w. lamp bank
95Ag 5Graphite (soft)	0.25	0.40	8000 w. lamp bank
93Ag 7Graphite (soft)	0.43	0.50	45 amp., resistance load
40Ag 60Ni..... (hard)	0.14	0.59	8000 w. lamp bank

shown in Table VIII. This information was prepared by the Electrical Laboratory of the Stackpole Carbon Co. The average contact resistance on a complete test is compared with the contact resistance calculated from Eq. 9 for the particular material. On these tests, the conditions varied widely from the ideal conditions assumed in the calculations since the surfaces were subject to arcing on opening and to considerable wear during the test. While there is one particularly wide deviation, the results are of the correct order of magnitude and appear to vary in the proper manner. In addition, the spread in the measured values of the contact resistance was quite wide in some cases, particularly where the contact resistance is high. A sufficient number of readings was taken, however, to give the averages their proper significance.

Since the surfaces were so far from the ideal, the question arises as to why the results are so consistent. The reason is not exactly clear, except that it is possible that variations in the value of *n* counterbalance the effects of the surface films. In any case, the choice of *n* as 1 would appear to give the results a sufficient agreement to be used as the safety factor for this particular relay. Another piece of test apparatus might require an entirely different value.

CONCLUSIONS

There are several important conclusions which can be drawn from the theory and experimental work presented above:

1. In general, the contact resistance force characteristic agrees with Eq. 9, except for the value of the constants.

2. The values of the constants in Eq. 9 are determined as much from the design of the apparatus (value of *n*) as from the contact materials and surface condition (value of *Y*).

3. The method of determining

the practical limit of contact force from Eq. 10 appears to be reasonably close to present practical experience.

4. A method is outlined for determining the maximum temperature rise to which particular contact materials may be subjected under atmospheric conditions.

5. A method is given for applying these temperature data to a determination of the contact drop which may be permitted on the contact material.

6. Based upon certain assumptions and tests of the maximum temperature rise permissible on contacts, it appears that this method may be applied to practice with reasonably good results.

To complete the data and to verify or disprove the results of these calculations, considerably more information should be gathered on the physical properties of the contact materials. The major holes in the experimental data are:

(*a*) Heat conductivities of contact materials,

(*b*) Maximum temperature which contact materials can stand under service conditions, such as arcing, continued opening and closing of the circuit, and under steady closed conditions, and

(*c*) Formation of surface films under various atmospheric and temperature conditions.

Acknowledgment:

The author cannot emphasize too strongly the help he has received from the various members of the A.S.T.M. Subcommittee X on Contact Materials, of Committee B-4. Their cooperation has furnished much of the experimental data presented here and their suggestions, criticisms, and encouragement have made this work possible. The individuals who contributed the experimental data have also given interesting and constructive criticism of the text. Further valuable comment was received from W. N. Goodwin, Director of Research, Weston Electrical Instrument Corp., and P. H. Dike, Assistant Director of Research, Leeds & Northrup Co., who were asked by the committee to review this work before publication. Appreciation is also expressed to the Management of the Stackpole Carbon Co. for permission to publish this work.

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DISCUSSION

Mr. N. Papazian[1] (*by letter*).—I have lately endeavored to gather theoretical as well as experimental data for bus contacts and have noted particularly the excellent paper by Mr. Shobert.

It is assumed that all of the equations (Eqs. 1 to 19, inclusive) in this paper with the proper substitution of constants can be applied to plain bar or tubular bus connections which are less complicated than less stable switching contacts.

In this connection, perhaps, it would be helpful to include aluminum and even wrought iron in the Tables I, II, and III of Mr. Shobert's paper and thus furnish a complete tabulation of constants for any kind of contact or bus connections.

Mr. Erle I. Shobert II (*author*).—The assumption that Eqs. 1 to 19 may be applied to plain bar or tubular bus connections is correct. Some understanding of the forces involved may be required to get accurate data, since the forces developed by bolts in bolted connections are not readily obtainable. In any case, however, the calculation should serve as a guide in applications of this type.

I have tried to find information concerning aluminum and wrought iron to add to the tables as suggested. Data concerning the physical properties are readily available on all types of aluminum and iron; however, the mechanical properties have such a wide influence upon the result that it would be necessary to know the hardness of the individual piece of material being used. This will depend upon its composition, cold working, and its entire previous history. I do not have enough information available at the present time to provide any reliable data for aluminum and wrought iron. I believe this is a case where some work should be done to investigate the effect of the physical properties of the various types of aluminum and wrought iron alloys and their treatment upon the contact resistance developed in bus connections. It would then be possible to decide which of the physical properties are important in these applications.

Mr. M. R. Swinehart[2] (*by letter*)—I believe that Mr. Shobert's paper presents an excellent theoretical background or basis on which to build empirical data for switch design.

In silver cadmium type contacts, I have known for some time that a given set would drop from an initial contact resistance millivolt reading of the order of 140 to about 100 after approximately one minute, dropping still further at a gradual rate to values of the order of 60 mv. after 30 min.

Although it was my original opinion that this decrease in contact resistance was caused primarily by decomposition of cadmium oxide occurring slowly at temperatures on the order of 900 C., I now believe that the initial and high rate of change in the first minute is caused by an increase in n, due to a decrease in Y (Eq. 9). This decrease in Y is probably caused by annealing.

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[1] Public Works Dept., Mare Island Naval Ship Yard, Vallejo, Calif.

[2] Development Engineer, Cutler-Hammer, Inc., Milwaukee, Wis.

The temperature rise of these contacts reaches a maximum value after about 15 min. and then shows a decrease through the remaining 15 min. of the test. I believe, after reading this paper by Mr. Shobert, that the gradual slope of the contact resistance curve following the initial one minute may be attributed to an increase in n caused by rubbing of the contacts as removable and stationary contacts heat up and expand, or, after 15 min., as they cool off again and contract, by more rubbing with additional increase in n.

Perhaps this rubbing action, caused by expanding and contracting contact mounting plates may explain the increase in n referred to in Mr. Shobert's paper in connection with the General Electric Test, Fig. 7.

MR. SHOBERT.—I appreciate Mr. Swinehart's comments very much. There are many effects on contacts which still have not been explained or described. Our committee is endeavoring to work the most important of these and has made considerable progress and any contributions which Mr. Swinehart may be in a position to make to our work will be greatly appreciated.

MR. S. G. ESKIN[3] (*by letter*).—Mr. Shobert's theoretical analysis seems to be basically sound, and with additional data the various constants could be brought in closer correlation with actual performance, thereby providing those designing electrical contacts, equations which would help a great deal in this difficult field.

In the conclusions, Mr. Shobert states that there is lack of data on heat conductivity of contact metals. The accompanying references[4] list sources where information could be obtained on heat conductivity of metals and alloys, in addition to the data listed in Table I of Mr. Shobert's paper. Also, one of the references is on thin films and may be of interest.

MR. SHOBERT.—The list of references given concerning the conductivity of contact metals is greatly appreciated. I am referring particularly to the heat conductivity of special alloys such as silver tungsten, silver nickel, and silver molybdenum materials. As far as I know, there is little data available on these special materials, although I have not yet gone through the complete list of references. I think that some of our activities should be directed toward methods of measuring these properties on these special materials.

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[3] Director of Research, Robertshaw-Grayson, Los Angeles, Calif.

[4] *References on heat conductivities of metals and alloys*:

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MECHANICAL METHODS OF TESTING*[1]

ANALYSIS OF THE STATE OF STRESS IN THE NECK OF A TENSION TEST SPECIMEN

By N. N. Davidenkov[2] and Miss N. I. Spiridonova[2]

In tension tests it has long been recognized as an advantage to consider true stresses s instead of the nominal stresses σ, obtained from the relationship of the deformation force to the final and not to the initial cross-sectional area of a specimen. However, the use of the term "true" in describing these stresses proved somewhat problematic since in plastic materials, in the formation of a neck, the distribution of stress in the section ceases to be uniform and "true" stress really proves to be only the average axial stress. There appear radial and tangential stresses, creating a triaxial condition of stress. This complicated condition of stress must be interpreted so that, on the basis of the tension test, it will be possible to determine the true properties of plasticity and strength of the material. Without such interpretation neither a correct evaluation of these properties nor a generalization of the obtained results as they apply to other states of stress (for example, torsion) is possible.

Unfortunately, no solution to this problem has been found as yet. There have been only a few scattered attempts which did not lead to undebatable results.

Thus, Koerber and Mueller (**1**)[3] studied the increase of the tensile strength (effect of strengthening due to shape) on models machined from uniformly deformed metal and they reproduced a neck in its various stages of deformation. They further applied this increase in the tensile strength to the process of concentrated deformation, ignoring the difference between the elastic and plastic distribution of stresses.

Druzhinin (**2**) machined a series of small specimens from the necks of specimens of a large diameter and in a tension test hoped to obtain a picture of the distribution of stress at fracture in the narrowest section of the specimen. However, the fractures appeared near the heads; that is, in the less deformed area beyond the limits of the neck itself, which made the results very indefinite.

Siebel (**3**) developed an equation of equilibrium for the curved elements of metal in the constricted zone (neck) but ignored the double curvature of these elements and, without any foundation, simplified his solution considering it as a plane problem in the meridional section of the specimen.

Only recently Bridgman (**4**) arrived at more accurate conclusions by taking into consideration the double curvature. At the same time he made an arbitrary assumption in regard to the circular form of the trajectories of the principal radial stresses (in meridional sections) which resulted in very complicated calculations yielding no reliable results. Both Siebel and Bridgman lacked ex-

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* Accepted for publication in *Proceedings*.

[1] Translated from Russian by A. Price for Westinghouse Research Laboratories, Pittsburgh, Pa., and the David Taylor Model Basin, Washington, D. C.

[2] Academy of Sciences of U.S.S.R., Polytechnic Inst., Leningrad, U.S.S.R.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1158.

perimental prerequisites that would serve as a basis for their theoretical calculations, without which the initial assumptions introduced into their calculations could not be justified.

We made it our aim to evolve the simplest, but experimentally based, theory of the distribution of stress in the smallest section of the neck of an elongated specimen (for one type of material) which would permit us in the first approximation to find the full tensor of stress for every point of this section. The only basis for an experimental approach was microscopic testing.

To determine the three principal stresses—axial s_l, tangential s_t, and radial s_r—the direction of which is determined by virtue of symmetry—it is essential to have three equations.

The first of these we derive by utilizing the known equation of plasticity (5):

$$\frac{s_l - s_t}{e_l - e_t} = \frac{s_l - s_r}{e_l - e_r} \quad \ldots\ldots\ldots (1)$$

where e_l, e_t, and e_r are the principal strains (true strains, that is, computed according to the formula $e = \int_{l_0}^{l} \frac{dl}{l}$, where l is the actual length of the deformed element). It is true that this equation has not been experimentally proven as fully accurate as was recently shown by Davis (6) but it can successfully be employed at least as a first approximation.

The second equation is derived from the general condition of the equilibrium of outer and inner forces in the section of the neck:

$$P = \int s_l dF \quad \ldots\ldots\ldots\ldots\ldots (2)$$

where P is the tensile force; F is the cross-sectional area (assuming that the law of change of s_l along the section is known). The third equation, giving the relationship between stresses s_l, s_t, and s_r, may be derived from the equilibrium condition of the stresses on an infinitesimally small element in the neck, cut out in the direction of the trajectory of the principal stress s_l. However, for the construction of this equation it is necessary to obtain certain relationships experimentally, the description of which now follows:

Armco iron was employed since it possesses all the needed qualities: high plasticity, single phase, and a precise microscopic outline of the grain boundaries (the composition of this iron being 0.04 per cent carbon, 0.04 per cent manganese, 0.01 per cent silicon, and 0.36 per cent copper). As an agent for etching the polished surface of the specimen a 5 per cent solution of picric acid in ethyl alcohol was used. For a sharper outline of the grain boundaries of the deformed metal it was necessary to repeat the process of polishing and etching the metal several times (sometimes as many as eight times). The extent of deformation of the specimen in various zones of the polished surface was determined by the average deformation of the grains in the field of vision of the microscope.

To measure the diameter of grains in a given direction a number of N grains was counted which crossed a straight line of a given length L, drawn on the frosted glass of the microscope (in our case 71.5 mm., magnification 100 times). Assuming that the average grains are of spherical shape, then the ratio of $\frac{N}{L}$ will give the size of the average length of the chord obtained by drawing a straight line through a series of variously distributed spheres. It may be shown (7) that with the increase of the number of spheres the length of this chord approaches $2D/3$, where D is the diameter of the sphere.

The same relationship is maintained for the ellipsoidal shape of grains crossing parallel to the main axis. However, instead of *D* the length corresponds to the main axis of the ellipsoid.

Thus the true average diameter of the grain is always 1.5 times larger than the measured chord. Hence, in order to simplify the calculations in determining the relative deformation, it is possible to calculate the chords by the above method without measuring the true diameter of the grain.

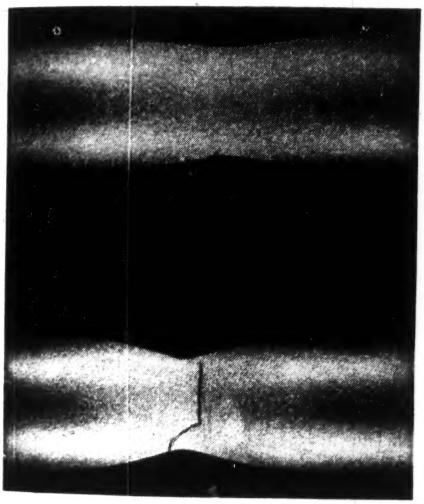

Fig. 1.—Specimens Nos. 5 and 6, Showing Various Shapes of Necks of Elongated Specimens.

First, it was necessary to be convinced that the grains of the specimen in its initial state have a sufficiently correct polyhedric shape and are free from any kind of systematic anisotropy. It was also necessary to measure the initial diameter of the grain (in the future, we will use this term for the above-mentioned "average chord"). A lengthwise and crosswise check of the polished surface of the specimen showed the length of the diameter to be 56.7 μ (the average diameter of the grain being 85 μ).

For further study eight specimens (25 mm. in diameter) were prepared and elongated to form necks of various dimensions. The data referring to these specimens are given in Table I and Fig. 1.

Specimens Nos. 3, 5, 8, and 6 of this series were subjected to microscopic inspection. With this aim in view, the specimens were cut crosswise at the narrowest section of the neck. A polished surface was prepared and annular zones were marked on it at various distances from the axis of the specimen as shown in Fig. 2. In each zone, at seven to eight points on the circumference, measurements were made of the diameter of the grains by the above-described method.

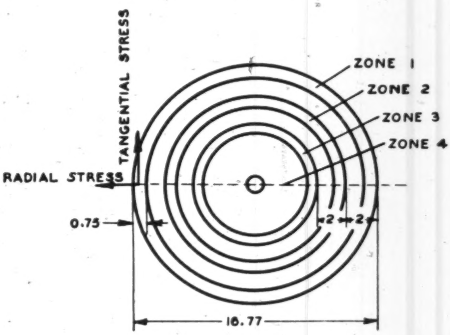

Fig. 2.—Zones of Microscopic Inspection of Specimen No. 3.

As an example, Table II shows the measurements obtained for specimen No. 5 in two directions, radial *r*, and tangential *t*.

Table III shows the measurements of grain diameters calculated by this method for three specimens, Nos. 3, 5, and 8. (It was impossible to make measurements of specimen No. 6 which fractured in the neck and because of the resultant damage to the structure manifested by numerous voids in the polished surface.)

The correctness of the measurements is confirmed by the agreement between

TABLE I.

Specimen	Diameter of Neck, mm.	Relative Contraction, ψ, per cent	True Contraction, per cent	True Stress s, kg. per sq. mm.	Radius of Curvature, R, mm.	Correction Factor, $1+\frac{a}{4R}$	Correction Factor, $\frac{R+0.5a}{R+0.25a}$
No. 3	19.18	41.0	54.2	49.6	95	1.025	1.02
No. 5	16.77	55.0	80.6	56.8	20.2	1.105	1.095
No. 11	16.66	55.8	82.4	58.5	21.35	1.098	1.09
No. 8	15.21	63.6	102.0	62.2	11.60	1.164	1.14
No. 12	14.81	64.2	107.3	67.2	11.46	1.160	1.14
No. 14	14.60	65.7	108.0	68.0	10.63	1.171	1.145
No. 6	14.04	68.4	116.8	69.8	8.25	1.213	1.175
No. 13	13.93	69.2	119.0	71.5	7.26	1.240	1.195

TABLE II.—NUMBER OF GRAINS IN LENGTH L = 71.5 mm. (× 100).

Measurement	Zone 1		Zone 2		Zone 3		Zone 4
	r	t	r	t	r	t	$r=t$
No. 1	18	18	18	18	19	18	20
No. 2	19	18	19	19	19	20	18
No. 3	19	20	19	19	18	18	18
No. 4	19	20	19	19	19	19	19
No. 5	17	18	18	19	19	18	18
No. 6	20	19	19	18	18	19	17
No. 7	18	18	19	20	19	19	19
N average	18.6	18.7	18.7	18.8	18.7	18.7	18.5
$\frac{N}{L}$ average	38.5	38.2	38.2	38.0	38.2	38.2	38.6
Average							38.3 μ

the deformation values calculated, on the one hand, on the basis of the general contraction of the specimen (ψ in Table I) and, on the other hand, on the basis of the deformation of grains (by means of the formula:

$$\psi = 1 - \frac{d^2}{d_0^2}$$

where d_0 is the size of the diameter in its initial state and d in the state of deformation) as can be seen from Table IV.

TABLE III.—SIZE OF AVERAGE DIAMETER OF GRAINS, μ.

Specimen	Zone 1		Zone 2		Zone 3		Zone 4	Average, μ
	r	t	r	t	r	t	$r=t$	
No. 3	44.2	44.6	44.6	44.2	44.6	44.6	44.6	44.5
No. 5	38.5	38.2	38.2	38.0	38.2	38.2	38.6	38.2
No. 8	34.5	34.7	33.9	33.8	33.6	34.1	33.7	34 0

An inspection of Tables II and III permits us to draw the following two extremely important conclusions:

1. The strain in the grains in the radial and tangential directions is practically the same and, therefore, on the basis of Eq. 1, the corresponding stresses are equal.

2. The strain does not change even in passing from zone to zone, that is, it is the same along the entire section of the

TABLE IV.

Specimen	Contraction ψ in per cent, determined by:	
	Measurement of the Neck	Measurement of the Grains
No. 3	41.0 55.0	38.5[a] 54.7
No. 8	63.6	64.0

[a] The lesser agreement may be explained by less accurate calculations because of larger sized grains.

specimen (for the given stage of development of the neck) and, therefore, the "effective" stresses are also the same. (We use the term "effective" to show the difference in the principal stresses, which difference is the cause for plastic deformation, according to hypotheses, of the maximum shearing stresses as well as of the octahedral shearing stresses.)[4]

It should be noted that Bridgman used both these deductions in his conclusions; he did so, however, without any experimental basis.

The second of the above-mentioned conditions, in particular, is subject to still another method of measurement, used as a check in our investigations: namely, microphotographs were taken of the polished surface. A square 5 by 5 cm. (at a magnification of 100) was outlined on these microphotographs and the number of grains that fell within its boundaries was counted.

TABLE V.

Specimen	Number of Grains in 5 by 5-cm. Square				
	Zone 1	Zone 2	Zone 3	Zone 4	Zone 5
No. 3	110	113	112	114	112
No. 5	158	154	160	156	...
No. 8	175	180	177	181	...

The obtained results are shown in Table V.

The fluctuations in the numerical results are within the limits of accuracy of the calculations and do not disclose any systematic changes along the radius of the specimen.

An attempt was made to measure the elongation of the grains lengthwise on the polished surface when cut out parallel to the axis of the specimen. However, these measurements were not accurate enough in view of the small number of grains falling within the field of vision and in view of the less defined outline of their cross boundaries. Actually there was no need for such measurements, since the law of constancy of volume permits us to determine the lengthwise elongation of the grains by their crosswise reduction.

Therefore, on the basis of the two above-mentioned deductions the third required equation can be set up to determine the stresses.

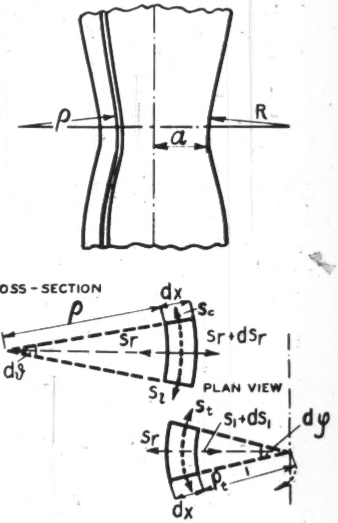

FIG. 3.—Diagram for the Equation of Equilibrium.

In inspecting the boundary curve of the neck as a trajectory of the principal stresses in the contour, we note that the curvature of the other trajectories must constantly decrease in size toward the axis, reaching the zero point on the axis of the specimen.

Let us take an element in the neck of the specimen (Fig. 3) along some intermediate trajectory having a radius of curvature ρ and a thickness dx along the radius. Let us define this element by two meridional planes of trajectory, forming the angle $d\varphi$; and by two planes, normal to the planes of the sketch, passing through the center of curvature

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[4] N. I. Prigorovski drew our attention to the fact that condition 1 is the geometrical deduction from condition 2, if it is proven for radial deformations.

O of the trajectory and forming angle $d\vartheta$.

Let us project on the radius all the forces acting upon this element and corresponding to the stresses shown in Fig. 3. The sum of all forces must equal zero. We derive the following equation:

$$-s_r\rho d\vartheta(x + dx)d\varphi + (s_r + ds_r)(\rho + dx)d\vartheta x d\varphi$$

$$- s_l dx\left(x + \frac{dx}{2}\right)d\varphi d\vartheta$$

$$+ s_l dx\left(\rho + \frac{dx}{2}\right)d\vartheta d\varphi = 0 \,..\, (3)$$

After reducing the equation by substituting s_r in place of s_l as previously proven, and dropping the infinitesimals of the second order, we arrive at the following equation after dividing by ρx:

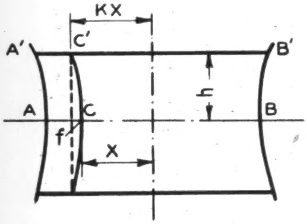

FIG. 4.—Diagram for Calculating the Curvature of Fibers.

$$ds_r = \frac{1}{\rho}(s_l - s_r)dx$$

Taking into account the constancy of the effective stress in the section, let us substitute constant s_0 for $(s_l - s_r)$ as a result of which the integration for point x will give:

$$s_r = s_0\int_x^a \frac{dx}{\rho} \,\ldots\ldots\ldots\ldots (4)$$

where a designates the radius of the section of the neck.

The relation of $\frac{1}{\rho}$ to x may be foreseen if the simplest assumption is made regarding the geometrical similarity of deformation in the sections near the smallest section of the neck. Let $A'B'$ be such a section at a distance h from AB (Fig. 4). Let us examine the fiber passing through point C at a distance x from the axis (a fiber which was originally straight) and let us assume that the other point of this fiber, lying within section $A'B'$, after deformation takes the position of C' at a distance kx from the axis, where k is some constant coefficient of proportion greater than 1.

TABLE VI.

Distance from axis x, mm.	Radius of Curvature ρ, mm.	Curvature $\frac{1}{\rho}$, mm.$^{-1}$
0	∞	0
0.8	62.8	0.0159
1.9	41	0.0244
2.625	28.5	0.0355
3.48	24.75	0.0404
5.14	14.6	0.0685
7.30	10.68	0.094

Thus we derive the following expression for the arrow of deflection f:

$$f = x(k - 1)$$

from which it further follows, assuming that the shape of the fiber is circular and that it has a radius of curvature ρ, that for small f:

$$h^2 = f\cdot 2\rho = x(k - 1)2\rho$$

and

$$\frac{1}{\rho} = \frac{2(k - 1)}{h^2}x$$

Therefore, the curvature is proportional to x and may be represented by formula:

$$\frac{1}{\rho} = \frac{x}{aR}$$

where a is the radius of the section of the neck and R is the radius of the curvature of its contour in the meridional section.

The correctness of this assumption was checked experimentally by measuring the

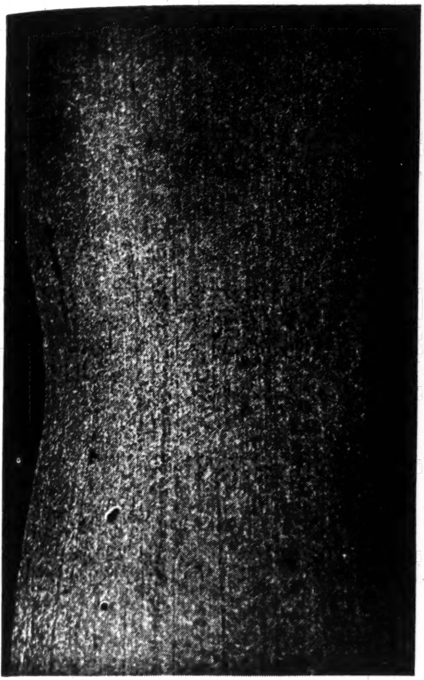

FIG. 5.—Lengthwise Polished Surface Through the Neck of a Specimen.

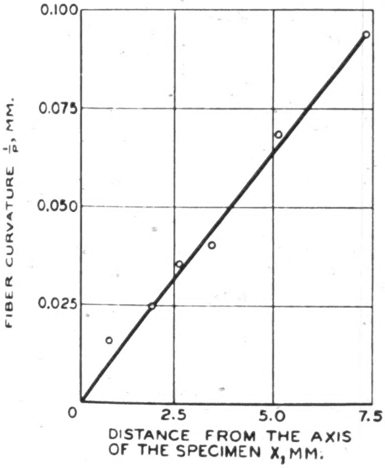

FIG. 6.—Change in Curvature of Fibers in the Neck of an Elongated Specimen.

curvature of fibers within the etched polished axial surface shown enlarged in Fig. 5.

This measurement at a number of points yielded the following figures shown in Table VI and in Fig. 6.

The linear relationship between x and $\frac{1}{\rho}$ stands out quite clearly here.

Now it is possible to make an integration of the Eq. 4:

$$s_r = s_0 \int_x^a \frac{x}{Ra}\, dx = \frac{s_0(a^2 - x^2)}{2Ra}$$

and

$$s_l = s_r + s_0 = s_0 \left(1 + \frac{a^2 - x^2}{2Ra}\right)$$

Once the relationship between $s_r = s_t$ and s_l is obtained, Eq. 2 may be used to find the absolute value of the stresses as follows:

$$P = \bar{s}_l \pi a^2 = s_0 \int_0^a \left(1 + \frac{a^2 - x^2}{2Ra}\right) 2\pi x\, dx$$

$$= \pi a^2 s_0 \left(1 + \frac{a}{4R}\right)$$

from which

$$\bar{s}_l = s_0 \left(1 + \frac{a}{4R}\right) \ldots\ldots\ldots (5)$$

where s_l is the average axial nominal stress in the section (values of factor $\left(1 + \frac{a}{4R}\right)$ are shown in Table I).

The greatest value of s_l in the section is obtained when $x = 0$

$$(s_l)_{\max} = s_0 \left(1 + \frac{a}{2R}\right)$$

$\bar{s}$ is related to the maximum s_l by equation:

$$(s_l)_{\max} = \bar{s}_l \frac{R + 0.5a}{R + 0.25a} \ldots\ldots\ldots (6)$$

(see last column of Table I).

The obtained distribution of the stresses s_l and s_r in the section of the neck is shown in Fig. 7. On the contour of the neck, where s_r is equal to zero, only the effective axial stress s_0 is active. In the remaining part, the axial stresses are distributed according to a parabolic law, repeating the distribution of radial stresses s_r and differing from them in the constant value of the effective stress s. The greatest shearing stresses $t_{\max}$ throughout the entire section are constant and equal to $\frac{s_0}{}$.

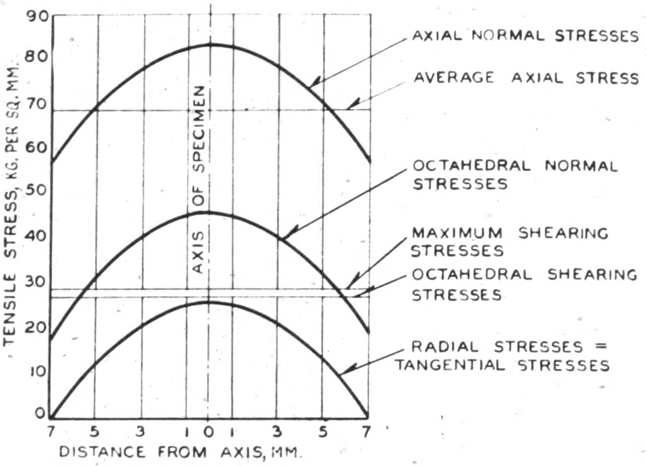

FIG. 7.—Distribution of Stresses in the Neck of Specimen No. 6.

The same diagram shows the octahedral (see Eq. 5) stresses (normal s_8 and shearing t_8), calculated on the basis of the formula:

$$s_8 = \frac{s_l + 2s_r}{3} = \frac{s_0}{3} + s_r$$

and

$$t_8 = \tfrac{1}{3}\sqrt{2(s_l - s_r)^2} = 0.47s_0$$

The curve of the octahedral normal stresses is parallel to that of s_r. These shearing stresses are constant throughout the section.

We will note that the obtained Eq. 5 expressing the increase in nominal stress s_l as against effective stress by means of the factor $\left(1 + \frac{a}{4R}\right)$, coincides with that arrived at by Siebel but has the advantage of a more rational theoretic basis and reliable experimental check.

TABLE VII.

$\frac{a}{R}$	Correction Factor Determined by	
	Bridgman	Siebel
0	1	1
1/3	1.078	1.083
1/2	1.115	1.125
1	1.215	1.250
2	1.386	1.500
3	1.524	1.750
4	1.649	2.000

Bridgman's formula, also without experimental basis, has the following complicated form:

$$s_l = s_0\left(1 + 2\frac{R}{a}\right)\ln\left(1 + \frac{1}{2}\frac{a}{R}\right)$$

It gives somewhat lower, although only slightly varying factors of value for s_0, as can be seen from Table VII.

Knowing the law of stress distribution in a neck, we can now apply the correction in the stress-strain curve based on the distorting effect of the neck.

For this purpose the necks of all eight specimens were photographed and by means of gradual selection the radii of curvatures of the outer contour at the narrowest section were found. These are shown in Table I. The same table shows the correction factors for the nominal stress.

With the aid of these figures it is now possible to construct a stress-strain diagram by taking into account the corresponding correction due to the distorting effect of the neck. It was of interest to construct this diagram on coordinates, permitting us to make a generalization for other types of states of stress and to compare it with the diagram obtained in a torsion test when the distorting effect of the neck is absent and when the condition of stress remains two-dimensional up to actual fracture.

With this aim in view a torsion test of two specimens made of the same material was conducted by constructing a diagram of the moments of torsion as a function of the angle of torsion and by calculating the greatest shearing stresses on the basis of the known Eq. (5):

$$t_{\max} = \frac{1}{2\pi r^2}\left(3M + \frac{\partial M}{\partial \theta}\,\theta\right)$$

Both diagrams of tension and torsion are constructed in Fig. 8 on one and the same coordinate scale. The tensile stress-strain diagram shows the maximum shearing stresses $t_{\max}$ equal to half of the effective normal stress s_0.

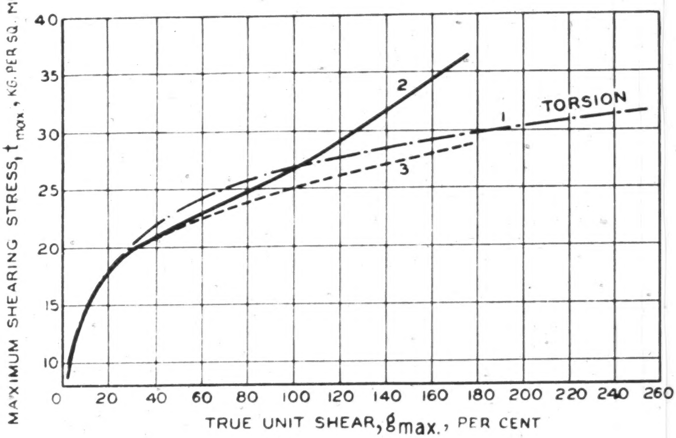

FIG. 8.—Comparative Curves for Tension and Torsion.
1 Torsion Curve.
2 Tension Curve Without Correction.
3 Tension Curve with Correction.

The unit shear is shown corresponding to the greatest true strains:

$$g_{\max} = e_l + e_r = 1.5e_l = 1.5 \ln \frac{1}{1 - \psi}$$

In the torsion diagram the greatest shearing stresses were calculated on the basis of the formula above referred to; and the unit shear $g_{\max}$ according to formula (8):

$$g_{\max} = \ln\left(1 + \frac{\gamma^2}{2} + \gamma\sqrt{1 + \frac{\gamma^2}{4}}\right)$$

where γ is the maximum unit shear, equal to the product of the radius of the specimen and the specific angle of twist.

The comparison of these curves in Fig. 8 was made on the basis of the theory of the maximum shearing stresses. For the purpose of comparison, the same diagrams were redrawn in Fig. 9 on the basis of the Huber-Mises hypothesis, developed by Nadai, using as coordinates the "octahedral shearing stresses t_8" and the "octahedral unit shears g_8." Calculations were made on the basis of the following formula (8):

$$t_8 = \tfrac{1}{3}\sqrt{2(s_l - s_r)^2} = 0.47s_0$$

$$g_8 = \tfrac{2}{3}\sqrt{2(e_l - e_r)^2} = 1.41e_l$$

An inspection of Figs. 8 and 9 will reveal that the tension and torsion curves, after the former has been corrected, almost coincide according to both methods of construction. In Fig. 8 the torsion curve lies above the tension curve for a portion of the graph, while in Fig. 9 the opposite is true. The true criterion for making a generalization with regard to these views concerning the stress conditions apparently lies somewhere in the middle and at the same time nearer to the hypothesis of the octahedral stress. It is interesting to note that in this way the main portion of the strain-hardening range is shown by the straight line portion of the tension curve. The material itself is strain-hardened much more slowly and, as can

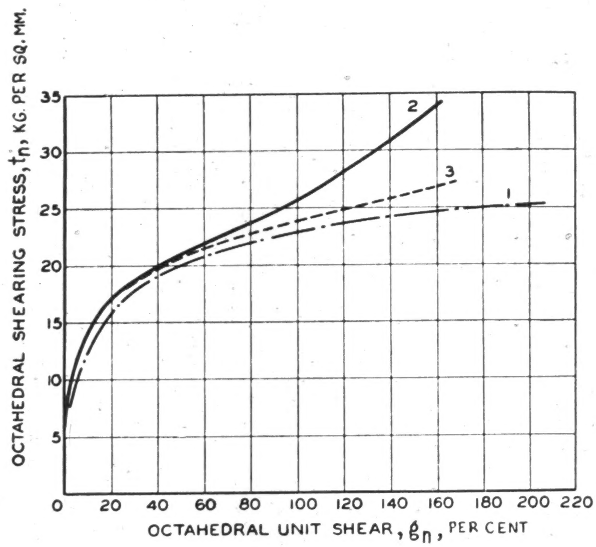

FIG. 9.—Comparative Curves for Tension and Torsion.
1 Torsion Curve.
2 Tension Curve Without Correction.
3 Tension Curve with Correction.

TABLE VIII.

	Specimen	$(s_l)_{max}$	t_{max}	t_8	g_{max}	g_8
Tension	No. 2	90.5	30.4	28.5	175	164
	No. 6	82.2	28.8 29.3	27.1 27.6	175 176	164 165
	No. 13	85.5	28.8	27.1	178	168
Torsion	No. *a*	31.4	31.4 31.3	25.6 25.5	253 229	207 187
	No. *b*	31.2	31.2	25.5	205	167

be well imagined, tends toward that state of saturation or deformation without much hardening that was observed by Taylor and Quinney (9) in their experiments with copper.

From the point of view of these experiments, there is no need even of making use of the theory of microfracture recently presented by Zener and Hollomon (10) for explaining the fact that the tension curve always lies higher than the torsion curve. As can be seen, this may be simply explained by the triaxial condition of stress in the neck.

For judging the conditions determining fracture, Table VIII shows the comparison of the stresses and strains at fracture for three tension and two torsion tests.

From Table VIII it may be concluded that the true resistance to fracture, expressed by the maximum shearing stresses, is 2 kg. per square millimeter less for tension than for torsion, but that expressed by the octahedral stresses is just the opposite—that much greater. If it is considered that this difference does not go beyond the limits of accuracy of all calculations, then it must be admitted that for tension as well as for torsion, fracture is caused by the shearing stresses and not by the normal stresses. This can clearly be seen from the first column in Table VIII as well as from the surface of the fracture itself, since the bottom of an ordinary "cup" of Armco iron consists of coarse elongated needles along the axis of the specimen. However, some effect of normal stress can still be discerned from the fact that $t_{\max}$ in all specimens is less for tension than for torsion.

Considering deformations we will note that the octahedral unit shears in fracture are more nearly similar to those obtained for tension than the maximum shears. Particularly for specimen "*b*" in Table VIII the unit shear almost coincides with that obtained in all three tension test specimens.

Thus, we have a method which permits us, by means of a microscopic test of polished surfaces, to obtain a more or less correct understanding of the true stress condition in the neck of the specimen and to find the relationship between the average stresses, as recorded by a testing machine and the true local stresses which allow us to determine the mechanical properties of the material. It now remains to apply this method to as great a number of plastic materials as possible and particularly to special steels, and each time to observe also the behavior of these materials in torsion so as to obtain a definite answer to the up-to-now unsolved problem regarding the true nature of the technical strength and concerning a generalization of these considerations from simple tension to complicated states of stress.

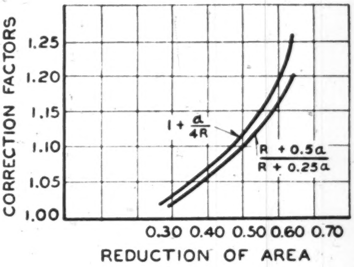

FIG. 10.—Diagram of Correction Factors.

Since there are no such experiments as yet, we can avail ourselves, in a first approximation, of the experimental fact that the shape of the neck, independently of the material, is determined by the "reduction of area" (calculated in relation to the area of the uniformly deformed portion F_B):

$$\psi_{\text{reduc.}} = \frac{F_B - F_k}{F_B}$$

where F_k is the cross-sectional area in the neck.

In Fig. 10 curves have been constructed giving the values of the factors $\left(1 + \dfrac{a}{4R}\right)$ and $\left(\dfrac{R + 0.5a}{R + 0.25a}\right)$, involved in Eqs. 5 and 6 as a function of this reduction of area.

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[See Joint Discussion, page 1175]

A STUDY OF THE TENSION TEST*

BY EARL R. PARKER,[1] HARMER E. DAVIS,[1] AND ALAN E. FLANIGAN[1]

SYNOPSIS

The paper describes some experiments and analyses that were made to determine the nature of the fracture in mild-steel cylindrical tension bars over a considerable range in temperature, to study the transition from a shear to a cleavage type of fracture in the simple tension specimen, and to obtain an indication of the state of stress which would account for the observation that shear type fractures originated in the central portion of a tension test specimen at normal temperatures.

Shear fractures were distinguished from cleavage (tension) fractures by microscopic examination of fractured ferrite crystals using an etch-pit technique. Tension specimens tested at 70 F. fractured entirely by *shear*, while at liquid-air temperature similar specimens failed entirely by *cleavage*. At intermediate temperatures a mixture of both types of fracture occurred.

An approximate analysis of the state of stress across the necked section of a tension bar just prior to rupture indicated that the shear stress was a maximum at the center of the necked section, corroborating the evidence from microscopic examinations which showed that the shear fractures started in the interior of the bars.

THE PROBLEM

The tension test has been employed for many years to indicate the suitability of materials for use in engineering structures. This test is one of the simplest of all tests to perform, it supplies accurate values for the yield strength and indicates the stress-strain characteristics of a material up to the strain at which necking begins. After the onset of local plastic flow, however, the stress-strain relationship becomes complex because of the curvature in the section. Also, circumferential and radial stresses are generated in this region. The stress condition gradually changes from one of simple uniform tension to a complicated condition of triaxial stress. The actual magnitudes and distribution of the stresses cannot be calculated from plastic theory with any certainty.

Approximate mathematical analyses of the state of stress in the necked section of a tension specimen just prior to failure have been made by a number of persons. One of the most recent is that of Bridgman (**1**)[2] which makes the simplifying assumption that the stress system is composed of simple axial tension upon which is superimposed a hydrostatic tensile component of stress which varies from zero on the surface to a maximum value at the axis of the bar. Bridgman's solution is in qualitative agreement with the opinions expressed by others concerning the prob-

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[1] Associate Professor of Physical Metallurgy, Associate Professor of Civil Engineering, and Research Engineer, respectively, University of California, Berkeley, Calif.

[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 1174.

able nature of the stresses in the necked section of a tension test specimen.

Observations by Ludwik (**2**) and others have demonstrated that cylindrical tension test specimens usually crack first near the axis of the bar, after which the crack propagates outward toward the surface. As a result of the evidence and opinions presented, several investigators (**2, 3, 4**) have stated that the crack which first forms near the axis of a test specimen is probably a cleavage[3] or tensile type of failure which occurs because of a condition of triaxial stress that exists at and near the origin of the crack. Thus the flat part of the common cup-cone fracture is often considered to be a tensile type of fracture, while the inclined portion or lip of the fracture near the surface is attributed to shear.

In recent years the so-called *true-stress:natural-strain*[4] curve has been frequently used to represent the results from the tension test (**5**). This representation of the stress-strain relationship has been used in the theory of plasticity as a basis for calculating the behavior of a metal at large plastic strains under various conditions of multiaxial stress. It is to be noted, however, that this relationship involves the *average* longitudinal stress and the *average* natural strain at the minimum section of a test bar. There is, then, some doubt as to the validity of predictions in the range of strain approaching failure because of the complex stress system which exists in the necked portion of the bar during the latter part of a tension test. The discrepancy, however, seems not to be excessive, so that for rough calculations the values of average true stress and average natural strain can be used. In a number of cases the correlations between results predicted from a true-stress:natural-strain curve and those actually obtained under multiaxial stress conditions have been fairly close.

The interest of the authors was aroused in this problem because of the possible error which might arise in connection with the use of the simple tension test to predict the behavior of the material under the action of certain multiaxial stress conditions, and because some observations made by the authors were not compatible with the statements recorded in the literature that the flat portion of the cup-cone fracture was a cleavage type of fracture caused by the high tensile stresses acting in this region.

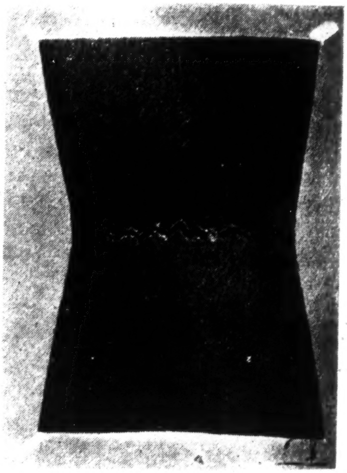

FIG. 1.—Path of Fracture Through Cylindrica Bar of Mild Steel Tested in Tension.

THE NATURE OF FRACTURE IN MILD STEEL

A close examination of the photographs in the literature (**2, 3, 4**) showing the first cracks which formed in the interior of tension bars and an examination of similar specimens prepared in the laboratory revealed that the apparently flat portion of the fracture was not really flat. As shown in Fig 1, the fracture traversed a zig-zag path and the actual planes of fracture were at approximately 45 deg. to the axis of the specimen rather than at 90 deg. This rather superficial evidence indicated that the fractures might be of the shear rather than the cleavage type. A more detailed study of the mechanism of fracture was considered necessary before definite conclusions could be drawn. Consequently, a microscopic examination of fractured specimens was made as described in the following paragraphs.

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[3] Sometimes called a "brittle" fracture. The following distinction is made: The term "brittle" is taken to refer to lack of ductility; the term "cleavage" is taken to refer to a physical mode of fracture, of the separation type, caused by tensile stresses. It is possible to obtain a cleavage fracture after considerable ductile action has occurred.

[4] True-stress = P/A, natural strain = $\log_e A_0/A$, where P is the load on the specimen at any strain, A is the actual minimum area sustaining this load, and A_0 is the original cross-sectional area of the specimen.

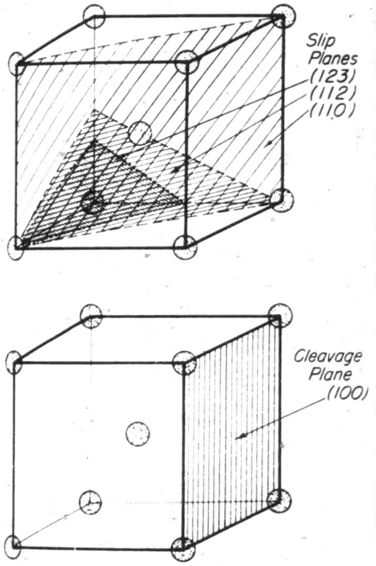

FIG. 2.—Slip and Cleavage Planes in Iron in Relation to Arrangement of Atoms in Crystal Lattice.

It had been previously demonstrated that in iron crystals the mechanism of shear deformation is one in which the slip planes slide past each other. It had also been demonstrated that a tensile or separation type fracture can occur in iron by cleavage in which the atomic planes are caused to separate by a tensile force, similar to the peeling apart of sheets of mica (6). Furthermore, in iron the shear planes and cleavage planes are not identical. Slip has never been observed on the cleavage planes and cleavage has never been known to occur on the slip planes. Figure 2 shows the location of the shear (or slip) planes and the cleavage planes in the iron lattice. To establish the mechanism of fracture in mild steel, it should be sufficient to identify the planes of fracture in the ferrite crystals. Because of these considerations, a medium-carbon steel was chosen for the investigation. It was found experimentally that a 20 per cent solution of nitric acid in water would produce etch pits in the iron crystals which would have sides parallel to the cleavage planes. Tension test specimens were then fractured at normal temperatures, sectioned longitudinally, electroplated with nickel to protect the fracture surface, and then polished and etched with the nitric acid solution. As shown in Fig. 3, the fractures in the centers of the specimens were found to follow paths which made angles of approximately 45 deg. with the sides of the etch pits (and also with the cleavage planes), and hence parallel to the slip planes in the ferrite crystals. Thus the mechanism of fracture was established for this case to be of a shear type.

For many years, it has been known that standard tension test specimens made of iron or mild steel will fracture in a brittle manner if tested at a sufficiently low temperature (7). Such specimens fracture after elongating only a few per cent, and no local necking occurs. The general surface of fracture is always normal to the axis of the specimen. To check the technique used for identifying the mechanism of fracture, tension specimens were fractured at various temperatures ranging from room to liquid-air temperatures, then the fractures were prepared and examined as described above. The fractured specimens are shown in Fig. 4. The fractures of specimens which failed in a brittle manner were found to follow the cubic planes of the ferrite crystals as shown in Fig. 5 and consequently were cleavage in nature. It was thus established that ferrite crystals can fail either by a shear or by a cleavage mechanism. The stress condition and the temperature determined the detailed mechanism of fracture. Some of the specimens fractured at intermediate temperatures were found to fail by cleavage in the central portion and by shear near the surface.

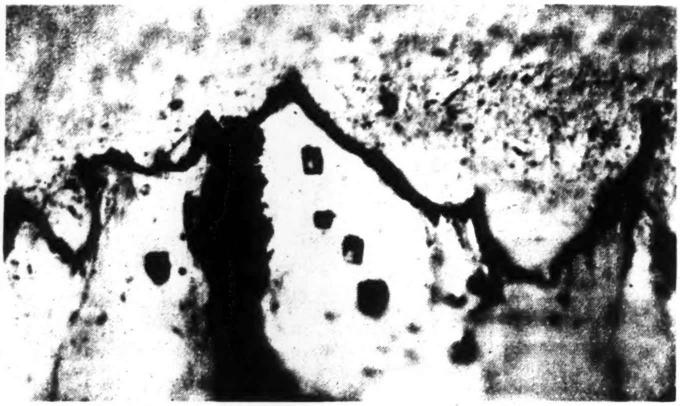

FIG. 3.—Photomicrograph of Section Perpendicular to Fracture Surface for a Shear Fracture (× 1500).

Specimen broken in tension at room temperature. Photograph taken at center of section where fracture originated. Traces of sides of etch pits in ferrite crystal are parallel to cleavage planes and are oblique to slip planes and to path of fracture through crystal.

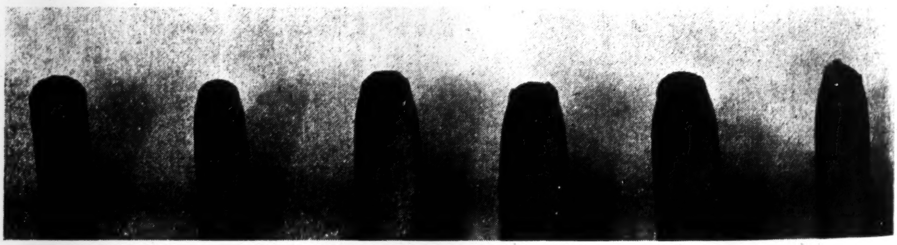

FIG. 4.—Fractured Tension Specimens of Mild Steel Broken at Various Temperatures.

As tests were performed at successively lower temperatures, the mode of fracture changed from shear to cleavage. At an intermediate temperature in the range shown, cleavage occurred at the center of a bar; at the lowest temperature, the entire fracture was by cleavage. The specimens tested at the two lowest temperatures shown were ⅜ in. in diameter instead of ½ in. in order to permit the use of more generous fillets at the end of the straight-sided portion of the bars.

INVESTIGATION OF STRESS DISTRIBUTION AT NECKED SECTION

The observation that ductile specimens tested at room temperature fracture by shear in the central portion of the specimen is not in accord with the expressed opinions of some investigators. Since the specimens did fail by shear, it seemed worth while to determine the reason for such failures. Two possibilities presented themselves. Either the shear stress was actually a maximum in the central part of the test specimens, which apparently is contrary to prevailing opinion, or else the shear fracture strength was influenced by the tensile stress acting across the slip planes. The only way of deciding which explanation is correct seemed to be to determine the state of stress in the interior of the specimens.

It is apparently not possible, from theoretical procedure thus far developed, to calculate accurately the exact state of stress in the necked portion of a tension test specimen. To obtain some idea of the stress distribution an experimental method of determining the approximate state of stress was devised. This method consisted of first straining in varying amounts a series of identical test specimens. These strains ranged from those too small to cause necking to those which which would almost cause fracture. The strained specimens were then unloaded, three longitudinal and three circumferential SR-4 resistance wire strain gages cemented to each specimen at the smallest part of the necked section, and the residual stress distribution in the necked section of the specimens determined by boring out the centers of the bars in successive increments and observing the strains on the surface as increasing amounts of metal were removed. From the surface strain measurements it was possible to calculate the approximate residual longitudinal, radial, and circumferential stresses which existed in the specimens prior to the boring operations. Then by superimposing upon the residual stresses the stresses calculated to have been relieved by the unloading of the bar before it was removed from the testing machine, it was possible to establish the approximate condition of stress which existed in the necked section of the specimen prior to the unloading.

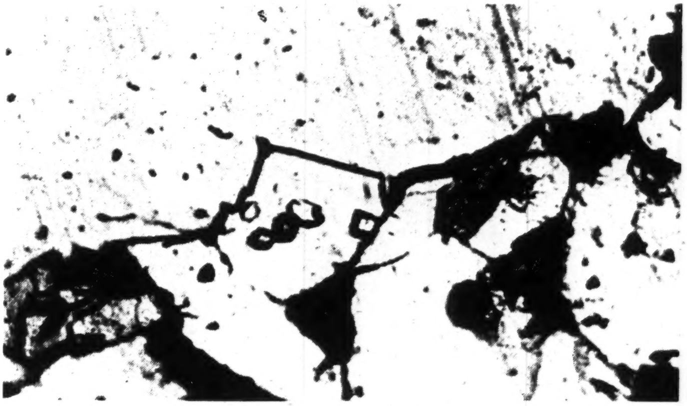

FIG. 5.—Photomicrograph of a Section Perpendicular to Fracture Surface for a Cleavage Fracture (× 1500).

Specimen broken in tension at liquid-air temperature. Traces of sides of etch pits in ferrite crystal are parallel to cleavage planes and to path of fracture through crystal.

The method of successive boring was developed by Sachs and Van Horn (8) to determine the residual stresses in long cylinders. This method assumes no variation in the stress condition over a considerable length of specimen, a condition which is not fulfilled in the necked tension specimens. Application of the method to necked tension specimens introduces some uncertainty as to the accuracy of the results. To check the magnitude of the error introduced by the use of the Sachs analysis for the residual stress determination in necked tension specimens, an additional experiment was performed. One tension specimen was strained almost to the breaking point and then unloaded. As with the other specimens, small resistance wire strain gages were placed at the minimum diameter section so that strains could be measured during machining. The specimens were normally bored in small increments, leaving the surface contour in its original condition. With the check specimen, however, the surface was first machined until the specimen was cylindrical, so that the geometry of the specimen was the same as that of the specimens used by Sachs. Strain measurements were made during the surface machining and also during the subsequent boring operations. The total strains measured during the machining of the specimen into cylindrical form were zero in the circumferential direction and 10 microinches per inch in the longitudinal direction. The total longitudinal strain resulting from the boring was over 800 microinches per inch and the average strain resulting from each of the fifteen or so separate boring steps exceeded 30 microinches per inch. The residual stress pattern determined from the specimen machined to a cylindrical shape was essentially the same as that obtained from a similar specimen having the necked contour. This experiment indicated that the magnitude of the error introduced by the shape of the necked specimens was of small magnitude.

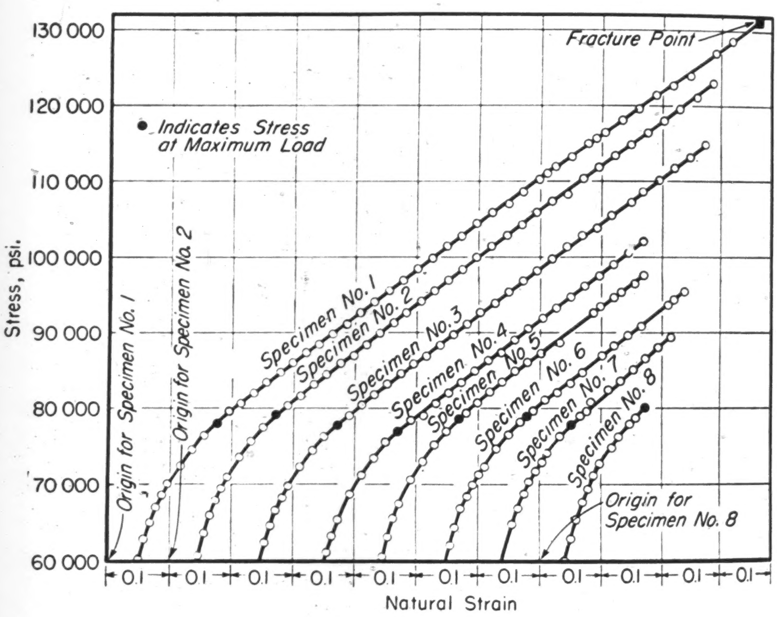

FIG. 6.—Diagrams of True Stress *Versus* Natural Strain for Specimens Used for Stress-Distribution Analysis.

Specimens were 2.25-in. diameter medium steel bars. Specimens were strained in tension to various amounts and then unloaded except specimen No. 1 which was tested to rupture.

In the calculation of the stresses due to unloading, it was necessary to consider the variations in stress due to the notching effect of the necked section. To estimate the stress-concentration effect, Neuber's analysis (9) for hyperbolic notches was used, and it was assumed that the unloading strains were elastic.

The Bauschinger effect was neglected

in the analysis. For higher magnitudes of strain in these tests, the Bauschinger effect is important and should be considered in a final analysis. During unloading of specimens subjected to large elongations, the axial stress at the surface changes from a high value in tension to a high value in compression. Because of the Bauschinger effect, yielding will occur during the latter part of the unloading operation. Due to yielding, the stress concentration which actually occurred was less severe than that indicated by the method of analysis which was used.

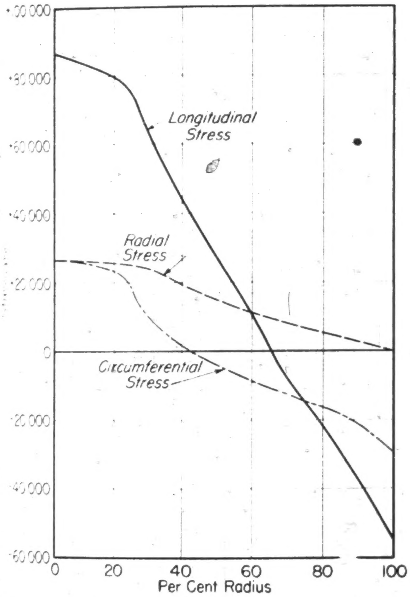

FIG. 7.—Calculated Distribution of Residual Stresses in a 2.25-in. Tension Bar Just Prior to Rupture.

Stresses were calculated for the conditions of strain at the point of rupture by extrapolation from the results of experimental stress analyses of bars strained to lesser amounts.

The material used for the investigation was a hot-rolled 0.25 per cent carbon steel having a yield strength of 47,500 psi., an ultimate strength of 68,500 psi., an elongation of 38 per cent in 2 in., and a reduction of area of 68 per cent as measured on a standard tension test specimen 0.505 in. in diameter. The specimens used for the stress-distribution determinations were 2.25 in. in diameter. This size was chosen so that the residual strain measurements could be made with a reasonable degree of accuracy. All test specimens were cut from a single length of bar and were given a stress-relief heat treatment of 2 hr. at 1100 F. before the tests were begun.

The true-stress:natural-strain curves for all of the specimens are given in Fig. 6. The natural strain was calculated from measurements of the diameter of the bar at the minimum cross-section after each increment of load, as indicated below:

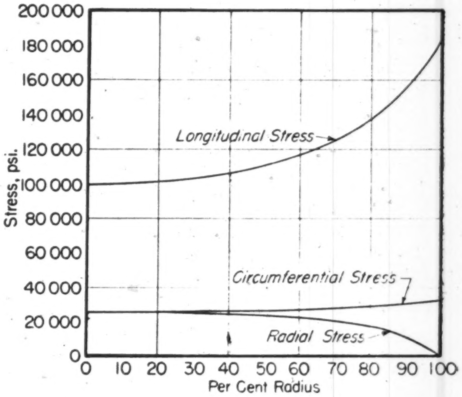

FIG. 8.—Calculated Distribution of Unloading Stresses in a 2.25-in. Tension Bar Just Prior to Rupture.

Stresses were calculated by the Neuber method of analysis for the shape of the bar at the point of rupture.

$$\text{Natural strain} = \int_{L_o}^{L} \left(\frac{dL}{L}\right)$$

$$= \log_e \frac{L}{L_o} = \log_e \frac{A_o}{A}$$

where:

L = final length of a small element,
L_o = original length of a small element,
A = final minimum cross-sectional area of test bar, and

A_o = original cross-sectional area of test bar.

The true stress was obtained by dividing the load by the minimum cross-sectional area of the specimen. After necking begins, this measure of true stress is only an average of the longitudinal stresses in the necked section. The actual stress distribution in this region is shown later.

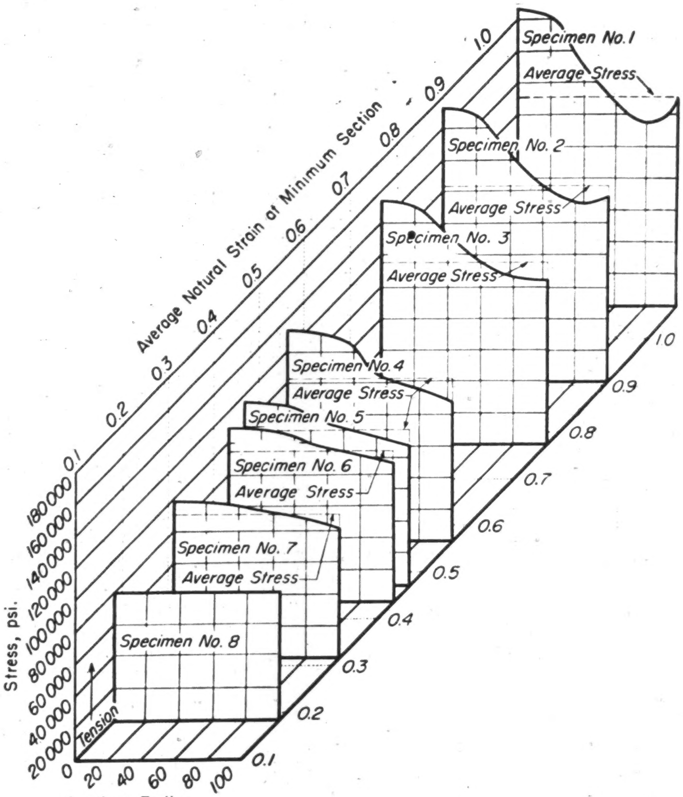

FIG. 9.—Distribution of Longitudinal Stresses at Minimum Section of Loaded Bars Strained to Various Amounts.

Diagrams are arranged according to the *average* natural strain attained under the given load applied to each specimen, see Fig. 6.

The strains to which the test specimens were subjected are shown in Fig. 6. The strained specimens were then unloaded, and three longitudinal and three circumferential SR-4 resistance wire strain gages were cemented to each of the specimens at the smallest part of the necked section. The residual stresses were then relaxed by boring out the central part of the specimen in successive increments. From the strains indicated by the strain gage readings taken during the boring operations, the approximate magnitude and distribution of the residual longitudinal, circumferential, and radial stresses which had existed in the bar just prior to the boring operation were calculated. The details of the procedure and methods of calculation are given in Appendix I. An example of a residual stress distribution obtained by this procedure is shown in Fig. 7.

As previously mentioned, the curva-

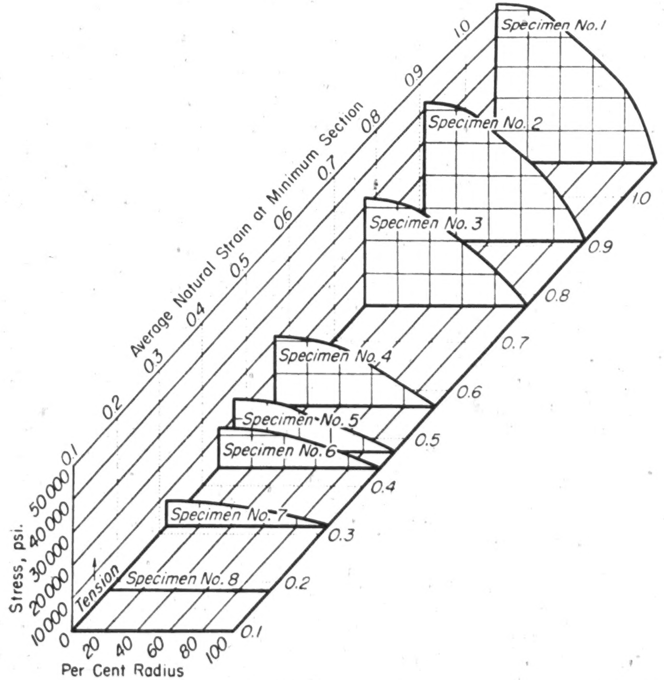

Fig. 10.—Distribution of Radial Stresses at Minimum Section of Loaded Bars Strained to Various Amounts.

Diagrams are arranged according to the *average* natural strain attained under the given load applied to each specimen, see Fig. 6.

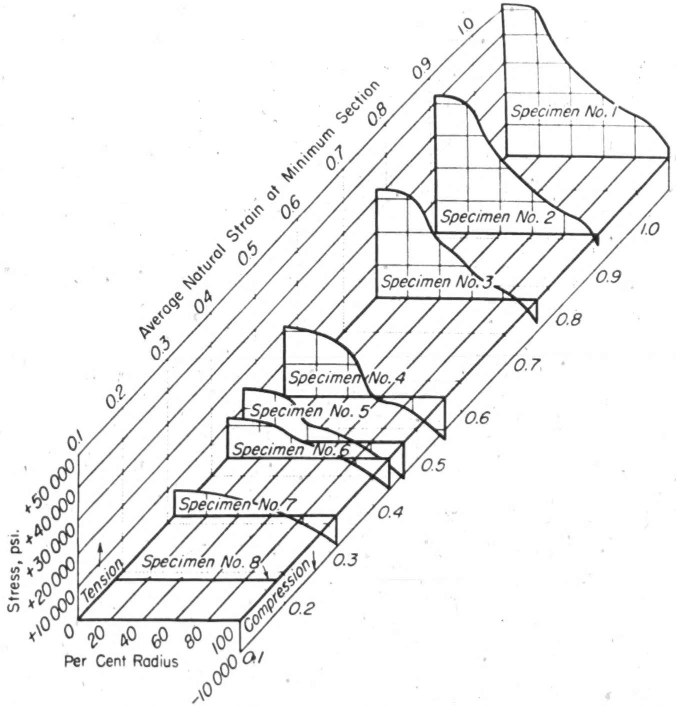

Fig. 11.—Distribution of Circumferential Stresses at Minimum Section of Loaded Bars Strained to Various Amounts.

Diagrams are arranged according to the *average* natural strain attained under the given load applied to each specimen, see Fig. 6.

tures in the necked sections of the specimens constituted notches which introduced mild stress-concentration effects in these bars. Furthermore, this circumferential notch introduced radial and circumferential stresses. The method of analysis devised by Neuber (9) permits the calculation of the magnitudes and distributions of the three components of stress in a bar with a hyperbolic notch for elastic strain conditions. Assuming that the unloading of a necked tension specimen is an elastic unloading procedure, the changes in the stresses may be considered to be the reverse of those produced by the elastic loading of a similar specimen having the same shape. An example of the results obtained by the Neuber analysis is given in Fig. 8. Some of the details of this analysis are given in Appendix II.

By adding the unloading stresses to the residual stresses, the longitudinal, radial, and circumferential stresses, under the maximum load sustained by each bar, at its minimum section were computed. The distributions of these computed stresses are shown in Figs. 9, 10, and 11. The stress-distribution diagrams in these figures are arranged according to the maximum natural strain developed by the bars. The stress distributions for specimen No. 1, which was actively loaded to rupture, were obtained partly by extrapolation from the data for bars subjected to loads smaller than the rupture load. Owing to the Bauschinger effect, the computed distributions of stress differ from the true distributions for the specimens subjected to the higher loads. However, a study of these results makes it evident that deviations from a uniform stress condition begin when necking begins and grow progressively greater as necking proceeds, and the data here presented give a qualitative picture of the effect of the necking on the stress distributions which are developed.

In Fig. 12, the circumferential, radial, and longitudinal stress distributions for specimen No. 1 are plotted on the diagram, together with the computed maximum shearing stresses. The shearing stresses were obtained from the differences in the major and minor principal stresses at each of a number of points across the section. *It is to be noted that the computed maximum shear stress occurs at the center of the section.* If the effects of plastic yielding, which occurs when compressive stresses are developed during unloading of a test bar (the Bauschinger effect), were considered, the difference between the shearing stresses at the center and at the surface of the bar would be somewhat greater than that from this analysis. This indicates then, at least qualitatively, why shear fractures can originate in the central portion of a necked tension bar.

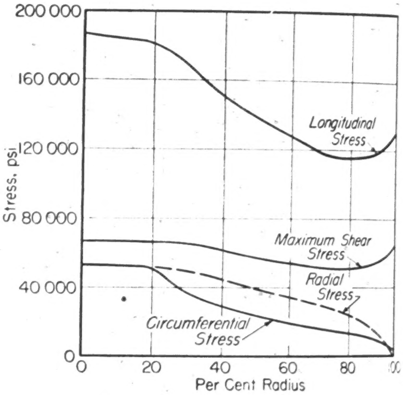

FIG. 12.—Calculated Distribution of Stresses Across Minimum Section of Necked Tension Bar at the Point of Rupture.

Stresses were obtained by summing the residual and unloading stresses for this condition.

It should be noted that the variation in shear stress across the necked section is small compared with the variation in longitudinal stress, and therefore the deviation of the actual shear stresses from the average shear stress is small. This may account for the approximate agreement between the results of many multiaxial stress tests and the results predicted from simple tension test data, because as long as shear is the controlling mechanism of failure, the computed average tensile strength is a function of the shearing resistance.

SUMMARY

1. It was established by microscopic examination that mild steel can fracture in two ways, either by shear or by cleavage. High shear stresses can cause shear fractures on slip planes within the ferrite crystals. Tensile stresses can cause cleavage on other crystallographic planes on which slip does not occur.

2. Shear-type fractures were found to occur across the entire section of rupture in ordinary tension test specimens of mild steel at normal temperatures; even the center portions of the cup-cone fractures were found to be of the shear type. At liquid-air temperature, only cleavage-type fractures were obtained. At intermediate temperatures, a mixture of both types of fracture occurred in some specimens.

3. The approximate magnitudes and distributions of the longitudinal, radial and circumferential stresses were determined in unfractured specimens which had been strained various amounts. The triaxial stresses generated in the necked section of a tension test specimen were found to be of such a nature that *the shear stress was greater at the center than at the surface.* This observation is in accordance with the fact that shear fractures begin in the interior of the specimen.

4. The variation in shear stress across the necked section was small compared with the variation in the tensile stress. Also, differences between the actual shear stresses and the "average" shear stress calculated from the average tensile stress were relatively small. This appears to account for the approximate agreement obtained by many investigators between results from the simple tension test and those obtained from multiaxial stress tests.

5. The reported stresses are only approximate because the Bauschinger effect was neglected. The results, however, give a useful picture of the stress distribution in the necked section of a tension test specimen.

Acknowledgments:

The authors wish to express their appreciation for the competent assistance rendered by E. F. McLaughlin and R. Payne in conducting the investigation.

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APPENDIX I

DETERMINATION OF RESIDUAL STRESSES AT NECKED SECTION OF 2.25-IN. TENSION BARS

A specimen of a ductile material subjected to tensile loading undergoes two stages of strain. While the load required to cause progressive extension increases gradually from zero to a maximum, the specimen undergoes continuous but uniformly distributed strain, some elastic and some plastic. At the maximum or ultimate load, necking begins and thenceforth essentially all of the plastic flow is restricted to a relatively small portion of the gage length. Continued straining results in a decrease in load even though the average stress in the necked section is greater.

The strains are not uniform throughout the necked region and a nonuniform distribution of longitudinal stress develops. At the same time, radial and circumferential stresses are induced in the region of the neck. Furthermore, the stress system induced in the necked section of the specimen stretched in tension is different from that which would occur in a bar machined to the same shape but loaded within the elastic range. In the machined specimen, the curvature in the region of the neck causes a concentration of stress at the outer surface, while the specimens strained in the plastic range to produce an identical shape has a maximum stress at the *center* of the necked section.

It is helpful to consider the stress system in a necked tension specimen as being composed of two parts: (1) a stress system resulting from removal of external load and (2) a superimposed residual or internal stress system not relaxed when the load is removed.

The residual stresses were determined experimentally by a method described by Sachs and Van Horn (8). This method consists of removing metal from the interior of the specimen by successive boring operations, measuring the resulting longitudinal and circumferential strains on the surface, and from these strains calculating the original stress distribution. All components of stress, the longitudinal, the radial and the circumferential, can be determined in this manner. The analysis is based on the assumptions that

$$\frac{\partial \sigma_c}{\partial \theta} = 0$$

$$\frac{\partial \sigma_L}{\partial L} = 0$$

where:

σ_c = circumferential stress,
σ_L = longitudinal stress,
θ = circumferential direction, and
L = axial direction.

The second of the assumptions mentioned above is, of course, only true at the minimum section. Consequently, the gage length used for the strain measurements must be small compared with the length of the necked portion of the specimen. This was accomplished by using type A7 SR-4 resistance wire strain gages which have gage lengths of approximately ¼ in.

The stresses at any location within the cross-section are related to the surface strains resulting from boring in the following manner:

$$\sigma_L = \frac{E}{1 - \mu^2}\left[(A_o - A_b)\,\frac{\partial(\epsilon_L + \mu\epsilon_c)}{\partial A} - (\epsilon_L + \mu\epsilon_c)\right]$$

$$\sigma_R = \frac{E}{1 - \mu^2}\left[\frac{(A_o - A_b)}{2A_b}\,(\epsilon_c + \mu\epsilon_L)\right]$$

$$\sigma_c = \frac{E}{1 - \mu^2}\left[(A_o - A_b)\,\frac{\partial(\epsilon_c + \mu\epsilon_L)}{\partial A} - \frac{A_o + A_b}{2A_b}\,(\epsilon_c + \mu\epsilon_L)\right]$$

where:

σ_L = longitudinal stress,
σ_R = radial stress,
σ_t = circumferential stress,
E = Young's modulus,
μ = Poisson's ratio,
A_0 = cross-sectional area before beginning boring operation,
A_b = cross-section of bore,
ϵ_L = longitudinal strain at surface, and
ϵ_t = circumferential strain at surface.

In the analysis, the cross-sectional area of the bore is plotted against the strain functions and the rate of change of strain with respect to the area is then obtained from these plots.

This analysis is rather complicated in nature, but appears to yield reasonably accurate results if certain precautions are observed. One important factor is the temperature. Extreme care must be taken to prevent the surface temperature of the specimen from rising more than 25 F. during the boring. A blast of air directed into the bore during machining helps to limit the temperature rise. It was found by experience that a temperature rise of more than 25 F. would cause abnormally high strain readings and would result in offsets in the bore area-strain curves. The cause of the error was not determined, although it may have been due to the introduction of machining stresses in the bore by the heavier cuts. In all boring operations, therefore, care was taken to limit the temperature rise of the specimen to a small magnitude.

When the radius of the bore approached the value of the radius of the section, the strain readings became very erratic. To check the effect of the boring operation itself on the strain readings, a stress-free specimen machined to the same shape as a necked specimen was bored in an identical manner. No strains were recorded until the wall of the specimen became thin. The strain readings then became erratic as in the case of the necked tension specimens. It was evident that the boring introduced errors in the strain readings only when the wall of the specimen became thin. The residual stresses could not be determined accurately for the last few per cent of the radius. However, the plotted values of cross-sectional area removed *versus* strain relaxed followed a smooth curve until the radius of the bore reached about 97 per cent of the outside radius, so that an extrapolation was considered sufficiently accurate to establish the stress condition at the surface.

APPENDIX II

CALCULATION OF UNLOADING STRESSES

As a necked tension specimen is unloaded, the average stress across the necked section is reduced to zero. Because of the shape of the necked portion, the actual magnitude of longitudinal stress relieved at the surface by unloading is not the same as that at the center. Furthermore, the radial and circumferential stress values are changed by the unloading. The determination of the stresses relaxed by the unloading thus involves a three-dimensional stress analysis. Neuber (9) has made an analysis by which there can be computed the three-dimensional stress distribution in a cylindrical specimen having a hyperbolic circumferential notch, when there exists proportionality between stress and strain. As shown in Fig. 13, the contours of the necked specimens could be closely matched to hyperbolas.

For convenience in his analysis, Neuber used a special quadratic coordinate system based upon the transformations

$$\left.\begin{aligned} x &= \sinh u \cos v \\ y &= \cosh u \sin v \end{aligned}\right\} \quad \ldots\ldots\ldots\ldots (1)$$

which yields an orthogonal set of coordinates. Then

$$\left.\begin{aligned} \left(\frac{x}{\sinh u}\right)^2 + \left(\frac{y}{\cosh u}\right)^2 &= 1 \\ \left(\frac{y}{\sin v}\right)^2 - \left(\frac{x}{\cos v}\right)^2 &= 1 \end{aligned}\right\} \quad \ldots . (2)$$

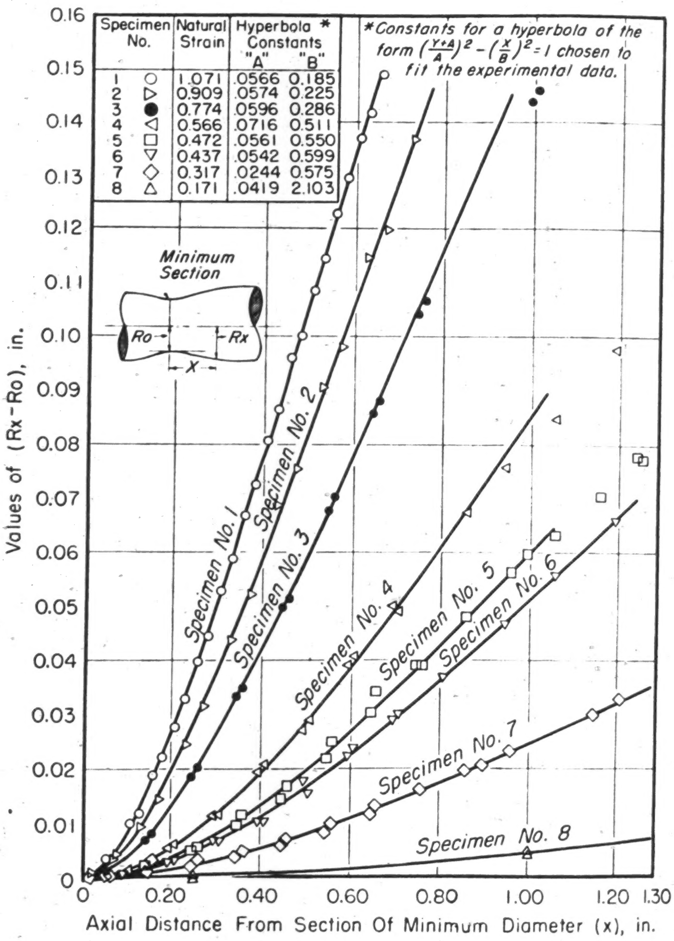

FIG. 13.—Contours of Necked Tension Bars Strained to Various Amounts.

In this system the lines, u = constant, are ellipses, and the lines, v = constant, are hyperbolas.

The equation relating the longitudinal stress to values of u, and the average stress is:

$$\sigma_u = \frac{1}{F^2}\left\{A \tanh^2 u + B\frac{\cos v}{\cosh^2 u} + C\left[-2 - D + \frac{1}{\cosh^2 u}\right]\cos v\right\} + \frac{\cos v}{F^4}(-A + B + C\cos^2 v) \ldots\ldots (3)$$

where:

σ_u = stress normal to ellipsoid u,
u, v = coordinates,
P = average stress on section,
A, B, C = constants depending upon contour of notch and upon P,
$D = 2(1 - \mu)$ where μ = Poisson's ratio, and
$F = \sqrt{\sinh^2 u + \cos^2 v}$.

At $u = 0$, the surface of the ellipsoid u collapses to the minimum cross-sectional area of the specimen and σ_u is then the longitudinal stress, σ_L. Also, at $u = 0$

$$\tanh^2 u = 0$$

$$\sinh u = 0$$

$$\cosh u = 1$$

$$F = \cos v$$

So

$$\sigma_L = \frac{1}{\cos v}\{B - C[1 + D]\} + \frac{1}{\cos^3 v}(-A + B + C\cos^2 v) \quad \ldots\ldots (4)$$

The constants can be evaluated from the following equations.

$$\left.\begin{aligned} A &= (D - 1)(1 + \cos v_o)C \\ B &= A - C\cos^2 v_o \\ C &= -\frac{P}{2}\,\frac{1 + \cos v_o}{1 + (2 - D)\cos v_o + \cos^2 v_o} \end{aligned}\right\} \ldots (5)$$

where P = average stress = load ÷ area of section, and v_o is the coordinate at the surface of the specimen. If Poisson's ratio is taken as 0.3, then $D = 1.4$.

The coordinate system was so chosen that

$$a/r = \tan^2 v_o$$

$$\frac{1}{\sqrt{a/r + 1}} = \cos v_o$$

$$\sqrt{\frac{a/r}{a/r + 1}} = \sin v_o$$

where:

a = radius of specimen at minimum section, and
r = radius of curvature at base of notch.

Hence

$$C = -\frac{P}{2}\,\frac{1 + \dfrac{1}{\sqrt{a/r + 1}}}{1 + \dfrac{0.6}{\sqrt{a/r + 1}} + \dfrac{1}{a/r + 1}}$$

from which the values for A and B follow directly.

The solution for σ_L thus consists of determining the values of P, a, and r experimentally, then substituting in Eq. 4 to determine the distribution of longitudinal stress across the section.

Similarly, the equation which relates the circumferential stresses in the minimum section to the coordinate, v, the average stress, P, the radius of the minimum section, a, and the radius of curvature of the notch, r, is

$$\sigma_c = \frac{1}{\cos v}\left(\frac{A}{1 + \cos v} - B - 0.6C\right) \quad \ldots\ldots (6)$$

where A, B, and C are the same constants used in Eq. 4.

The radial stress is given by

$$\sigma_R = \frac{1}{\cos v}\left\{\frac{-A}{1 + \cos v} - 0.6C\right\} + \frac{A - B}{\cos^3 v} \quad . (7)$$

The analysis as given above is based on the assumption that the notch is very deep. Neuber has made a similar analysis for shallow notches and, by a combination of these two analyses, he has arrived at a solution for the concentration effect of notches of intermediate depth. The stress-concentration factor used by Neuber for shallow notches is

$$\alpha_S = \frac{\sigma_{\text{max.}}}{\sigma_{\text{ave.}}} = 1 + 2\sqrt{t/r} \quad \ldots\ldots (8)$$

where:

t = depth of notch, and
r = radius of curvature at base of notch

As an example of the difference in the stress-concentration factor obtained for specimen 1 in the necked tension bar series, the deep notch theory gives a value for α of 1.45 while the shallow notch theory results in values of α_S ranging from 1.40 to 1.44, depending upon the value chosen for t. The difference in the maximum stress is obviously small. The necked tension specimens appear to be more nearly in the shallow notch class and consequently a corrected value of α of 1.41 was used as the most probable stress-concentration factor.

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JOINT DISCUSSION

MECHANICAL METHODS OF TESTING

By N. N. Davidenkov and Miss N. I. Spiridonova

A STUDY OF THE TENSION TEST

By Earl R. Parker, Harmer E. Davis and Alan E. Flanigan

Mr. A. Nadai.[1]—It seems a fortunate coincidence that two valuable papers are offered for presentation at this annual meeting of the Society, originating from two countries, devoted to the same subject, in which new experimental evidence is presented for obtaining quantitative information regarding the distribution of the stresses in the constricted portion of bars of ductile material before they break in tension tests. Although the determination of the true stresses at which a tension specimen breaks is one of the oldest mechanical problems the testing of materials has raised since its earliest times, it has always been considered as a question an answer of which encounters formidable difficulties. The reasons are familiar to most engineers. Before a bar of a ductile material breaks it deforms locally and plastically by large amounts, making it necessary to introduce precise means for measuring the permanent strains and for defining the stresses under which these latter are produced in the necked portion of the specimen or, in other words, to analyze a space distribution of stress and strain in three dimensions. Both papers approach this problem from an experimental angle, proposing different means for the determination of the maximum stresses at which the fracture starts in the minimum section of the bar. Both papers are based on theoretical considerations, the assumptions of which may be more or less debatable. This writer does not wish to question any of these assumptions because he is also of the opinion that simplifying assumptions must be made in order to obtain, with a minimum of theoretical means, quantitative results regarding the distribution of the stresses at fracture. While this writer, therefore, does not wish to raise various additional important factors which according to his own opinion may have an influence on the form of the necked portion of a bar, he does wish to submit one or two additional remarks. The problem of the necking is admittedly a rather complex problem in the testing of ductile materials. A satisfactory complete analytical solution of the necking problem of the ductile metals thus appears very difficult for many reasons, some of which have been quoted in the two papers and some by previous investigators of the necking problem. In the range of the strain hardening of the metals, the mean true stress during a tension test increases with the mean axial strain. It has not yet been established definitely whether—and if so, how much—the permanent deformation influences the strength of a polycrystalline grain structure before the fracture occurs. Some experimental evidence seems to make it probable that small elements of material which have been distorted plastically in some prescribed manner may become gradually anisotropic with regard to their inner resistance against separation at the instant when fracture occurs. Expressed in other words, the inner resistance against separation after a considerable amount of cold work may be different in the different directions relative to the material elements.

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[1] Consulting Mechanical Engineer, Westinghouse Research Labs., East Pittsburgh, Pa.

The writer suspected for some time[2] and believes still that a mechanical theory of necking of ductile metals in tension which is based on the concept of pure strain hardening is unable to predict one important group of observations related to necking, namely, the great differences in the shapes of the profiles of the necked portions of tension specimens which have been observed when these were tested under different rates of straining (or at slightly different temperatures). It is well known that specimens tested slowly at slightly elevated temperatures show much longer necks with gradually changing diameters than bars broken by rapid pulling. In these latter cases the constriction is much more localized and shorter at the instant of fracture. Two Russian investigators, A. A. Ilyushin[3] and A. J. Ishlinsky[4] have recently worked out a mathematical theory of necking assuming certain idealized conditions, namely, a material which has a definite yield point and which, after this has been reached, deforms like a viscous material (in which stress is proportional to the permanent rate of strain). They found the very interesting result that bars of such an ideal material having a profile with shallow waves may or may not neck further down in the narrowest sections. This depends on the rates with which the bars are stretched. If the rate is below a certain rate, the shallow waves are wiped out through further stretching. If the rate is above the critical value, the waves will become deeper and deeper and necking will proceed in the bars.

The authors of both papers referred to theoretical considerations concerning the necking of round bars.[5] In these, the assumption was made more recently that the radial and tangential unit strains are equal to each other in the minimum section of the bar. It has furthermore been assumed that these strains are perfectly constant through the minimum section. According to the theory of plasticity this has the consequence that in every point of the minimum section the radial stress must become equal to the tangential stress. The radial stress is 0 at the surface of the neck, therefore also the circumferential stress is 0 in the surface. At the axis of the specimen, in the center of the minimum section, on the other hand, the radial stress equals the circumferential for symmetry reasons. It was the merit of Davidenkov and Spiridonova of having proved in their paper through their careful measurements that the distortion of the grains in ingot iron in the necked portion of the specimens indeed conforms with this assumption in a satisfactory manner. Thus, the assumption that $\epsilon_r = \epsilon_t$ is verified by their experiments with this metal. Such special modes of flow disclosing a striking and beautiful symmetry condition cannot occur without some deeper cause of a mechanical nature. The writer wishes to point to a possible cause for this type of symmetrical flow $\sigma_r = \sigma_t$.

This will be illustrated through a figure of the surface of yielding. Attention to this type of flow has been called by F. K. Th. van Iterson in a little book which has just been published in Paris, "Traité De Plasticité pour L'Ingénieur," 1944, p. 98. Van Iterson states "that among all possible states of stress satisfying a given condition of plasticity in nature those occur which make the efforts (forces) a minimum."

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[4] A. J. Ishlinsky, "Stability of the Plastico-Viscous Flow of a Round Bar," *Journal*, Vol. 7, No. 2, pp. 109-130 (1943); also W. W. Sokolovsky, "The Theory of Plasticity—An Outline of Work Done in Russia," *Journal of Applied Mechanics*, Am. Soc. Mechanical Engrs., Vol. 13, No. 1, p. A1 (1946).

[5] P. W. Bridgman, *Transactions*, Am. Soc. Metals, Vol. 32, p. 553 (1944).

It can be shown that this symmetrical type of flow indeed produces a minimum value of the tensile force which is the integral of the axial stresses taken over the minimum section in the neck of a specimen among other possible types of distributions of stress also satisfying the plastic limit.

Suppose that the surface of yielding is expressed by the condition

$$(\sigma_r - \sigma_t)^2 + (\sigma_t - \sigma_a)^2 + (\sigma_a - \sigma_r)^2 = 2\,\sigma_0^2$$

where σ_0 is the yield stress for pure tension in an advanced state of strain hardening of the metal approaching the last stages of stretching just before the fracture may occur. Assuming that σ_a, σ_r, and σ_t (the axial, radial, and tangential stress at the distance r from the axis in the minimum section) are plotted as rectangular coordinates (see accompanying Fig. 1) this is the equation of a circular cylinder whose axis makes equal angles with the σ_a, σ_r, σ_t directions. On the right of the cylindrical surface of yielding is shown a portion of a necked bar in two views. The testing engineer wishes to express the distribution of the axial stresses σ_a within the minimum section (as indicated in the middle figure through the curve for σ_a) of the neck.

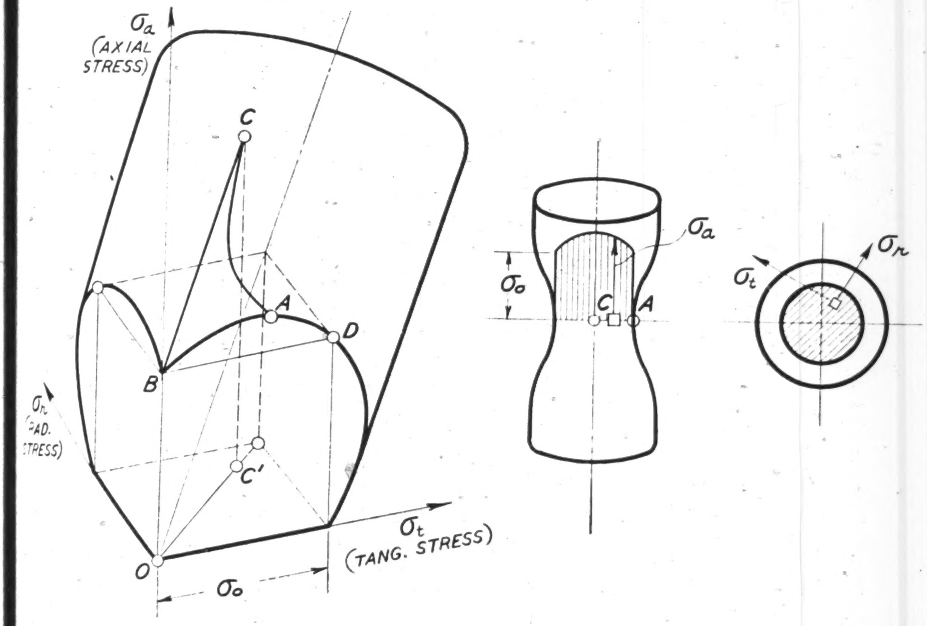

FIG. 1.—Surface of Yielding.

The cylindrical surface of yielding intersects the coordinate planes in two ellipses shown in the figure. To point *A* of the bar (at the surface of the neck) must correspond a point *A* situated on one of these ellipses namely in the plane section of the surface of yielding in which $\sigma_r = 0$ (since the radial stress σ_r must vanish in point A on the surface of the tension specimen). At point C, the center of the minimum section (or in the axis of the tension specimen), σ_r equals σ_t. The corresponding point C (representing the state of stress) on the surface of yielding must evidently be situated in the plane bisecting the right angle of the σ_r and σ_t axes (since in this plane $\sigma_r = \sigma_t$). Thus we see that all states of stress in the minimum section of the tension specimens will be represented by a curve which connects the points A and C on the cylinder. This curve has been traced in the accompanying Fig. 1 on the cylindrical surface of yielding. The ordinates σ_a of this latter curve (such as CC') represent then the axial stresses σ_a of the bar in its minimum section just before fracture occurs.

It is now easy to see that the integral of all these ordinates:

$$P = 2\pi \int_0^a \sigma_a r dr$$

which is the load at fracture *becomes a minimum* if the curve CA is made to coincide with the steepest straight line CB which can be traced on the cylinder, namely the generatrix passing through C. All other curved lines traced on the cylinder starting from point C and joining a point A in the σ_a, σ_t plane have longer ordinates σ_a than the straight generatrix CB. But along this latter

$$\sigma_r = \sigma_t$$

Thus we may state that according to the theory of flow of a perfectly plastic substance a ductile metal adjusts itself to flow under tension in such a manner that in the minimum cross-section in the neck everywhere the radial stress becomes equal to the circumferential stress because under these symmetrical states of stress the bar can flow *under the minimum value of the tensile force*, which otherwise could make it also flow if the σ_r would not be equal to the σ_t.

It is remarkable to note that the two different experimental methods which were proposed in the two papers lead their authors to a distribution of the stresses at fracture which is similar in one important respect: the tangential stress practically vanishes at the surface of the neck in the minimum section in the curves given by Parker, Davis, and Flanigan during the last stages of stretching conforming with what Davidenkov and Spiridonova concluded from their measurements of the strains. The differences between the shapes of the stress curves at earlier stages of stretching may also be of interest for further studies.

A STUDY OF THE GEOMETRY OF THE TENSION-IMPACT SPECIMEN[*],[1]

By N. A. Kahn[2] and E. A. Imbembo[2]

Synopsis

Data are presented on the effect of the geometry of the tension-impact specimen on the energy values obtained from the tension-impact test. This paper indicates the effect of the L/D ratio of the test section on the tension-impact values and includes recommended values for this ratio in order to obtain a true measure of the tensile-impact resistance of metallic materials, as may be determined by tests conducted in a conventional pendulum type impact testing machine. Tension-impact data for medium steel, austenitic type corrosion-resisting steel, copper, Naval rolled brass, nickel-copper alloy (monel), and 17S-T type aluminum alloy at various L/D ratios are included.

Considerable confusion exists in the literature (**1, 2, 3, 4, 5**)[3] on tension-impact testing to the extent that various investigators employ different forms of test specimens and the results of tests are not always expressed in the same units and terminology. For example, several investigators (**2, 3**) have expressed the results of tension-impact tests in terms of the number of foot-pounds required to rupture specimens of certain dimensions; another (**4**) has reported results in terms of unit energy absorption (foot-pound per cubic inch of metal contained in the gage section); while still another (**5**) has indicated values in terms of so many foot-pounds per square inch of cross-sectional area of the gage section. A situation of this kind obviously does not permit satisfactory correlation of the data of various workers in the field. While the authors make no special claims for the tension-impact test conducted at relatively low velocities, such as are available in pendulum type impact testing machines, the work described herein was undertaken in an effort to throw further light on the subject and to provide data which can be used to effect standardization of this test. However, the tension-impact test does afford a simple and quick determination of toughness which is sometimes measured by the more laborious method of taking the total area beneath the stress-strain diagram (to rupture) as obtained in the static tension test.

In November, 1943, the authors were authorized to conduct a metallurgical investigation of welding-quality medium steel plate intended for hull construction for the purpose of obtaining data which would serve as a basis for specification improvement and quality control. Among the various properties to be

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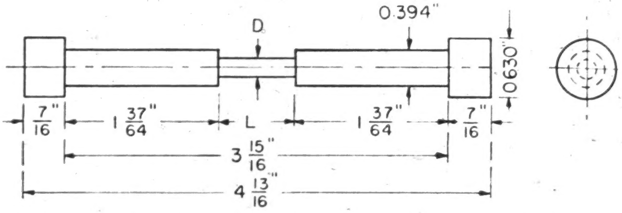
[*] Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] The opinions or assertions contained herein are the private ones of the authors and are not to be construed as official or reflecting the views of the Navy Department or the Naval Service at large.

[2] Principal Metallurgist and Senior Metallurgist, respectively, Material Laboratory, New York Naval Shipyard, Brooklyn, N. Y.

[3] Boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1190.

TABLE I.—EFFECT OF *L/D* RATIO ON THE TENSILE IMPACT RESISTANCE OF MEDIUM STEEL RIVET ROD.



¾-in. Diameter As-Rolled Bar Stock. Tests Conducted at Room Temperature Under a Striking Velocity of 16.5 ft. per sec.

Gage Length, *L*, in.		0.05	0.10	0.25	0.50	1.0
GAGE DIAMETER, *D* = 0.113 IN.						
L/D		0.442	0.885	2.21	4.42	8.8
Volume, cu. in.		0.0005	0.001	0.0025	0.005	0.0
Total Energy Absorption, ft-lb.	No. 1	3.0	3.25	4.5	11.5	16.0
	No. 2	2.75	4.0	4.5	8.5	...
	No. 3	3.0	3.5	5.5	10.5	14.5
	No. 4	2.75	3.5	5.25	7.75	15.7
	No. 5	2.75	3.5	5.5	8.0	15.0
	Avg.	2.85	3.55	5.05	9.25	15.3
Unit Energy Absorption, ft-lb. per cu. in.	No. 1	6000	3250	1800	2300	1600
	No. 2	5500	4000	1800	1700	...
	No. 3	6000	3500	2200	2100	1450
	No. 4	5500	3500	2100	1550	1575
	No. 5	5500	3500	2200	1600	1500
	Avg.	5700	3550	2020	1850	1530
GAGE DIAMETER, *D* = 0.160 IN.						
L/D		0.313	0.625	1.56	3.13	6.2
Volume, cu. in.		0.001	0.002	0.005	0.01	0.0
Total Energy, Absorption, ft-lb.	No. 1	3.75	7.0	11.75	23.25	35.0
	No. 2	5.5	10.0	11.0	24.0	35.0
	No. 3	5.0	8.0	13.5	24.0	40.0
	No. 4	8.0	7.0	11.5	23.75	37.0
	No. 5	4.75	7.75	13.0	24.25	44.5
	Avg.	5.40	7.95	12.15	23.85	38.3
Unit Energy, Absorption, ft-lb. per cu. in.	No. 1	3750	3500	2350	2325	1750
	No. 2	5500	5000	2200	2400	1750
	No. 3	5000	4000	2700	2400	2000
	No. 4	8000	3500	2300	2375	1850
	No. 5	4750	3875	2600	2425	2225
	Avg.	5400	3975	2430	2385	1915
GAGE DIAMETER, *D* = 0.195 IN.						
L/D		0.256	0.513	1.28	2.56	5.1
Volume, cu. in.		0.0015	0.003	0.0075	0.015	0.0
Total Energy, Absorption, ft-lb.	No. 1	6.0	13.25	20.5	37.0	66.7
	No. 2	8.0	...	20.0	38.0	55.2
	No. 3	6.75	13.75	23.0	35.0	53.5
	No. 4	8.0	12.0	19.75	37.25	53.2
	No. 5	8.5	11.5	22.0	33.25	75.5
	Avg.	7.45	12.63	21.05	36.10	60.8
Unit Energy, Absorption, ft-lb. per cu. in.	No. 1	4000	4420	2740	2460	2225
	No. 2	5330	...	2680	2530	1840
	No. 3	4500	4580	3060	2340	1780
	No. 4	5330	4000	2640	2480	1770
	No. 5	5660	3830	2930	2220	2520
	Avg.	4965	4210	2810	2405	2030
GAGE DIAMETER, *D* = 0.226 IN.						
L/D		0.221	0.442	1.11	2.21	4.42
Volume, cu. in.		0.002	0.004	0.010	0.02	0.04
Total Energy Absorption, ft-lb.	No. 1	12.75	15.25	29.5	51.25	103.0
	No. 2	14.5	17.25	30.25	49.25	83.0
	No. 3	14.5	16.0	29.5	48.0	106.5
	No. 4	15.0	16.75	30.0	48.5	84.0
	No. 5	15.0	18.25	29.5	48.5	85.5
	Avg.	14.35	16.70	29.75	49.1	92.4
Unit Energy Absorption, ft-lb. per cu. in.	No. 1	6375	3810	2950	2560	580
	No. 2	7250	4310	3025	2465	080
	No. 3	7250	4000	2950	2400	660
	No. 4	7500	4190	3000	2430	100
	No. 5	7500	4560	2950	2430	140
	Avg.	7175	4175	2975	2455	2310
GAGE DIAMETER, *D* = 0.252 IN.						
L/D		0.198	0.397	0.992	1.98	3.97
Volume, cu. in.		0.0025	0.005	0.0125	0.025	0.05
Total Energy, Absorption, ft-lb.	No. 1	22.25	20.5	37.0	63.5	105.0
	No. 2	26.0	26.0	42.0	65.0	116.0
	No. 3	22.75	19.0	37.5	59.0	118.5
	No. 4	23.5	23.5	37.5	61.0	118.5
	No. 5	20.75	20.5	39.5	60.5	120.0
	Avg.	23.05	21.90	38.7	61.8	115.6
Unit Energy, Absorption, ft-lb. per cu. in.	No. 1	8 900	4100	2960	2540	2100
	No. 2	10 400	5200	3360	2600	2320
	No. 3	9 100	3800	3000	2360	2370
	No. 4	9 400	4700	3000	2440	2370
	No. 5	8 300	4100	3160	2420	2400
	Avg.	9 220	4380	3095	2470	2310

studied was the behavior of this material when subjected to tensile-impact loading. In this regard, the authors were confronted with the problem of selecting an appropriate design for the tension-impact specimen which, as far as the authors are aware, has not as yet been fully standardized.

In approaching the problem, cognizance was taken of the work of Mann (4) and of the fundamental fact that the energy absorbed in rupturing a metal in tension impact is a function of the volume of metal participating in the absorption of energy, which volume is determined by the length of the uniform section of least diameter. The problem was therefore resolved into determining the proper relationship between the diameter of the least section and its length, which would provide for a true measure of the tensile-impact resistance of the material.

MATERIALS AND TESTS

In December, 1937, the authors completed a series of tests (similar to those made by Mann (4)) using a specimen of the form shown at the top of Table I. Numerous combinations of gage diameter D and gage length L were tried in an effort to learn the effect of the geometry of the test section on the tensile-impact resistance of a medium steel. The material used for this purpose consisted of $\frac{3}{4}$-in. round hot-rolled medium steel rivet rod conforming to Navy Department Specification 43-R-1. The tests were made at room temperature (70 to 80 F.) in a 120 ft-lb. Amsler impact testing machine at a striking velocity of 16.5 ft. per second corresponding to the maximum available kinetic energy of the pendulum. It is to be noted that this velocity is appreciably below the transition velocity, the existence of which was well demonstrated by Mann (6).

When the present problem arose, the data obtained in 1937 were brought to light and studied for any salient features. The results of these tests are given in Table I and shown graphically in Fig. 1, in which values of unit energy absorption (obtained by dividing the total energy by the volume of metal corresponding to the length of the least diameter section) have been plotted against ratios of L/D. As suggested by Mann (4), this curve indicates the presence of three zones; for L/D ratios of less than approximately 2, a zone of energy concentration, increasing to very high values with decreased L/D ratio; for L/D ratios between approximately 2 and 5, a zone of nearly constant energy absorption; and for larger values of L/D, a zone of gradual decrease in the unit energy absorption.

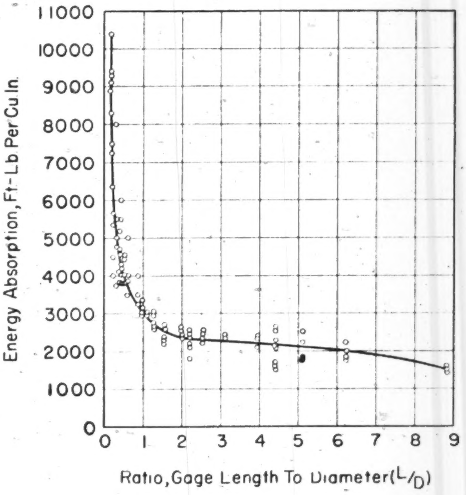


FIG. 1.—Effect of L/D Ratio on Unit Energy Absorption Values Obtained in Tension-Impact Tests of Medium Steel Rivet Rod, As-Rolled.

Tests conducted at room temperature. Striking velocity, 16.5 ft. per sec.

The existence of a zone of high energy concentration may be explained on the basis that specimens with very short gage lengths show apparently higher tensile strength values because of the

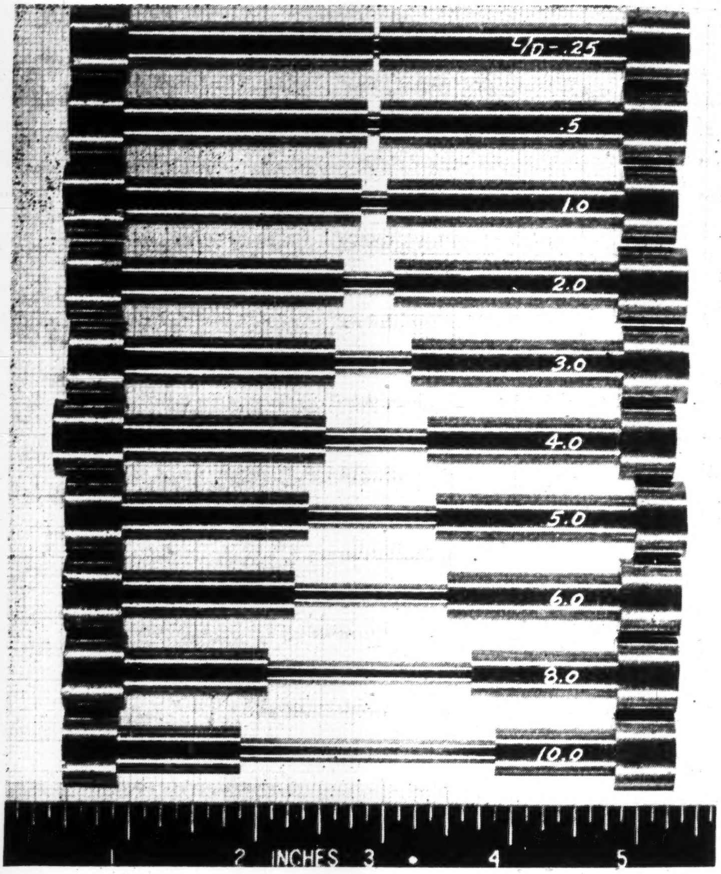


FIG. 2.—Tension-Impact Specimens of Copper with Various L/D Ratios in the Gage Section.

TABLE II.—CHEMICAL AND PHYSICAL CHARACTERISTICS OF ADDITIONAL MATERIALS SELECTED FOR TENSION-IMPACT TESTS.

	Copper	Naval Rolled Brass	Nickel-Copper Alloy (Monel)	Corrosion-Resisting Steel (18-8)	Aluminum Alloy (17S-T
Navy Dept. Specification	47C2	46B6	46M7	46S18	46A4
Bar stock size	¾-in. D	¾-in. D	⅞-in. D	¾-in. D	1⅛-in. D
Condition	Annealed at 750 F.	Annealed at 1000 F.	Annealed at 1500 F.	Annealed	Heat treated
Copper, per cent	> 99.99	60.00	31.47	....	4.18
Zinc, per cent	....	39.19	...	....	....
Tin, per cent	....	0.79		....	....
Iron, per cent	....	....	1.19	....	0.75
Aluminum, per cent	....	....	0.09	....	Remainder
Carbon, per cent	....	....	0.18	0.04	....
Manganese, per cent	....	....	0.60	0.70	0.60
Chromium, per cent	....	....	....	18.46	Si—0.39
Nickel, per cent	....	....	66.32	8.89	Mg—0.51
Proof stress, psi	....	Insufficient Material for Static Tension Test	40 000	67 500	....
Yield point, psi	10 500		42 500	....	43 130
Tensile strength, psi	32 880		90 000	116 380	65 250
Elongation in 2 in., per cent	56.0		44.5	37.5	23.0
Reduction of area, per cent	70.1		72.7	67.7	38.5

strengthening effect of the shoulders. While specimens with very short notch lengths are capable of absorbing relatively large amounts of energy, as indicated by the unit energy values, it will be observed from Table I that the total energy values are small because of notch effect. The decrease in unit energy for *L/D* ratios greater than approximately 5 is probably due to the fact that the phenomenon of necking is a localized effect; for specimens of long gage length, the entire length (or volume) of the gage section does not fully participate in the absorption of energy since the ends of the gage section do not undergo much deformation.

TABLE III.—RESULTS OF TENSION-IMPACT TESTS OF COPPER, ANNEALED AT 750 F.

Tests conducted at room temperature under a striking velocity of 16.5 ft. per sec.

Nominal *D*, mm.	Nominal *L*, mm.	*L/D*	Total Energy, ft-lb.		Gage Volume, cu. in.	Unit Energy, ft-lb. per cu. in.	
			Individual Values	Average		Individual Values	Average
5	1.25	0.25	13.5 15.0	14.25	0.0015 0.0015	9 000 10 000	9500
5	2.5	0.5	13.8 15.0	14.4	0.0030 0.0030	4 600 5 000	4800
5	5.0	1.0	16.5 18.0	17.25	0.0060 0.0060	2 750 3 000	2875
5	10.0	2.0	23.5 23.8	23.65	0.0119 0.0119	1 970 2 000	1985
5	15.0	3.0	31.0 31.3	31.15	0.0179 0.0179	1 730 1 750	1740
5	20.0	4.0	40.0 40.0	40.0	0.0238 0.0238	1 680 1 680	1680
5	25.0	5.0	46.3 48.0	47.15	0.0299 0.0298	1 550 1 610	1580
5	30.0	6.0	56.5	56.5	0.0356	1 590	1590
5	35.0	7.0	59.3 66.0	62.65	0.0418 0.0421	1 420 1 570	1495
5	40.0	8.0	66.3	66.3	0.0477	1 390	1390
5	45.0	9.0	67.5 81.0	74.3	0.0537 0.0532	1 260 1 520	1390
5	50.0	10.0	78.0 79.0	78.5	0.0602 0.0600	1 300 1 320	1310

It was considered desirable to confirm the results obtained on medium steel by conducting a series of similar tests on other common metals. For this purpose, the materials described in Table II were selected. The copper, Naval rolled brass, and nickel-copper alloy were first homogenized by suitable annealing treatments. The corrosion-resisting steel and the aluminum alloy, as drawn from stock, were in the annealed and heat-treated conditions, respectively. Specimens of the form and dimensions shown in the sketch at the top of Table I were prepared with various *L/D* ratios from each of these materials. A typical set of machined specimens is illustrated in

TABLE IV.—RESULTS OF TENSION-IMPACT TESTS OF NAVAL ROLLED BRASS, ANNEALED AT 1000 F.

Tests conducted at room temperature under a striking velocity of 16.5 ft. per sec.

Nominal *D*, mm.	Nominal *L*, mm.	*L/D*	Gage Volume, cu. in.	Total Energy, ft-lb.	Unit Energy, ft-lb. per cu. in.
5	1.25	0.25	0.0015	8.0	5330
5	2.5	0.5	0.003	10.0	3330
5	5.0	1	0.006	17.5	2920
5	10.0	2	0.012	27.0	2250
5	15.0	3	0.018	40.5	2250
5	20.0	4	0.024	50.0	2080
5	25.0	5	0.030	65.0	2170
5	37.5	7.5	0.045	87.0	1930
5	50.0	10	0.060	117.5	1960

TABLE V.—RESULTS OF TENSION-IMPACT TESTS OF NICKEL-COPPER ALLOY (MONEL), ANNEALED AT 1500 F.

Tests conducted at room temperature under a striking velocity of 16.5 ft. per sec.

Nominal *D*, mm.	Nominal *L*, mm.	*L/D*	Total Energy, ft-lb.		Gage Volume, cu. in.	Unit Energy, ft-lb. per cu. in.	
			Individual Values	Average		Individual Values	Average
5	1.25	0.25	17.3 18.5	17.9	0.0015 0.0015	11 530 12 330	11 930
5	2.5	0.5	21.5 22.0	21.75	0.0030 0.0030	7 170 7 330	7 250
5	5.0	1.0	29.0 29.3	29.15	0.0060 0.0059	4 830 4 970	4 900
5	7.5	1.5	37.5	37.5	0.0091	4 120	4 120
5	10.0	2.0	47.5 47.5	47.5	0.0119 0.0119	3 990 3 990	3 990
5	12.5	2.5	58.5	58.5	0.0153	3 820	3 820
5	15.0	3.0	61.5 69.0	65.3	0.0178 0.0180	3 460 3 830	3 645
5	17.5	3.5	77.5	77.5	0.0207	3 740	3 740
5	20.0	4.0	85.5 87.5	86.5	0.0236 0.0239	3 620 3 660	3 640
5	22.5	4.5	99.8	99.8	0.0269	3 710	3 710
5	25.0	5.0	106.3 108.5	107.4	0.0296 0.0300	3 590 3 620	3 605
5	27.5	5.5	116.3	116.3	0.0329	3 540	3 540

Fig. 2. The results of tension-impact tests have been tabulated in Tables III to VII inclusive and plotted in Figs. 3 to 7 inclusive, respectively. A graphical summary of the results is shown in Figs. 8 and 9. Figure 10 represents a photograph of the ruptured Naval brass specimens.

TABLE VI.—RESULTS OF TENSION-IMPACT TESTS OF ANNEALED CORROSION-RESISTING STEEL (18-8).

Tests conducted at room temperature under a striking velocity of 16.5 ft. per sec.

Nominal *D*, mm.	Nominal *L*, mm.	*L/D*	Total Energy, ft-lb.		Gage Volume, cu. in.	Unit Energy, ft-lb. per cu. in.	
			Individual Values	Average		Individual Values	Average
5	1.25	0.25	13.5	13.5	0.0015	9000	9000
5	2.5	0.5	19.0	19.0	0.0030	6330	6330
5	5.0	1.0	31.0	31.0	0.0058	5350	5350
5	7.5	1.5	41.0 43.0	42.0	0.0090 0.0091	4560 4730	4645
5	10.0	2.0	53.3 53.5	53.4	0.0118 0.0120	4520 4460	4490
5	12.5	2.5	63.0 63.0	63.0	0.0152 0.0149	4140 4230	4185
5	15.0	3.0	76.5	76.5	0.0180	4250	4250
5	17.5	3.5	88.0	88.0	0.0209	4210	4210
5	20.0	4.0	101.5	101.5	0.0242	4190	4190
5	22.5	4.5	110.5 120.0	115.3	0.0270 0.0272	4090 4410	4250

TABLE VII.—RESULTS OF TENSION-IMPACT TESTS OF HEAT-TREATED ALUMINUM ALLOY (17S-T).

Tests conducted at room temperature under a striking velocity of 16.5 ft. per sec.

Nominal *D*, mm.	Nominal *L*, mm.	*L/D*	Total Energy, ft.-lb.	Gage Volume, cu. in.	Unit Energy, ft-lb. per cu. in.
7	1.75	0.25	14.0	0.0040	3500
7	3.5	0.50	14.0	0.0083	1690
7	7.0	1.0	27.5	0.0163	1690
7	10.5	1.5	37.5	0.0247	1520
7	14.0	2.0	46.0	0.0325	1420
7	17.5	2.5	56.0	0.0412	1360
7	21.0	3.0	65.5	0.0494	1330
7	28.0	4.0	82.5	0 0658	1250
7	35.0	5.0	102.8	0.0821	1250

It is to be noted that the unit energy absorption curves indicated in Figs. 3 to 7 inclusive are essentially of the same form as that shown in Fig. 1 and that the zone of constant unit energy absorption generally begins at an *L/D* ratio of

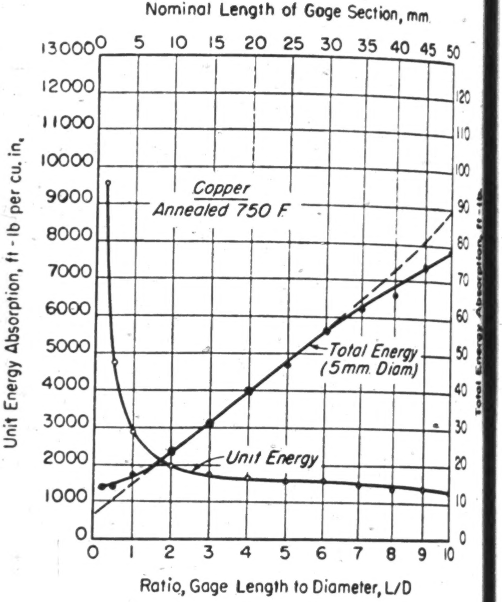


FIG. 3.—Effect of *L/D* Ratio on Unit Energy Absorption Values Obtained in Tension-Impact Tests of Copper, Annealed at 750 F.

Tests conducted at room temperature. Striking velocity 16.5 ft per sec.

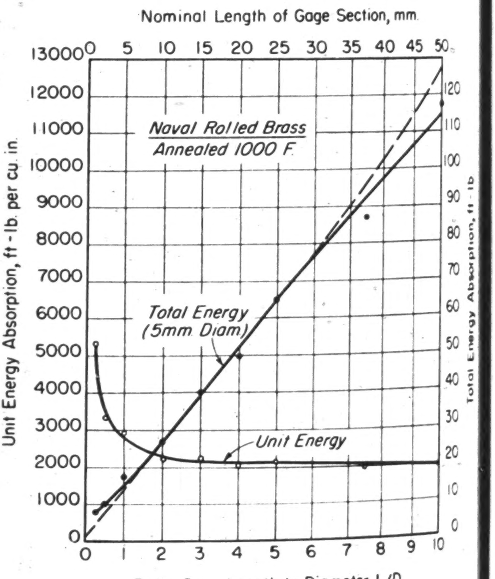


FIG. 4.—Effect of *L/D* Ratio on Unit Energy Absorption Values Obtained in Tension-Impact Tests of Naval Rolled Brass, Annealed at 1000 F.

Tests conducted at room temperature. Striking velocity, 16.5 ft. per sec.

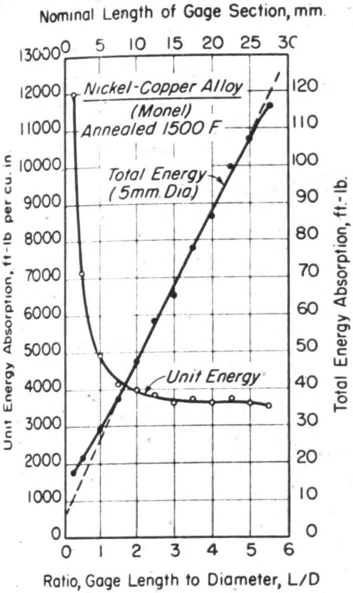


FIG. 5.—Effect of L/D Ratio on Unit Energy Absorption Values Obtained in Tension-Impact Tests of Nickel-Copper Alloy (Monel), Annealed at 1500 F.

Tests conducted at room temperature. Striking velocity, 16.5 ft. per sec.

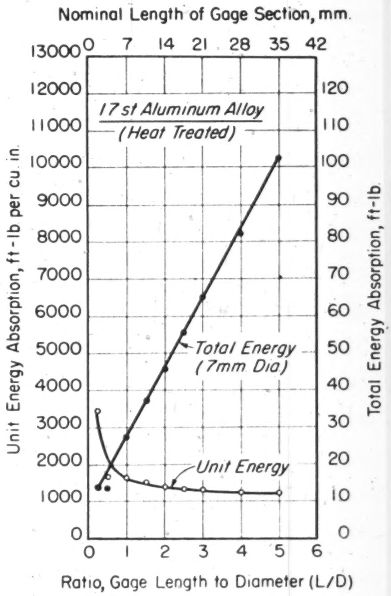


FIG. 7.—Effect of L/D Ratio on Unit Energy Absorption Values Obtained in Tension-Impact Tests of 17S-T Aluminum Alloy, Heat-Treated.

Tests conducted at room temperature. Striking velocity, 16.5 ft. per sec.

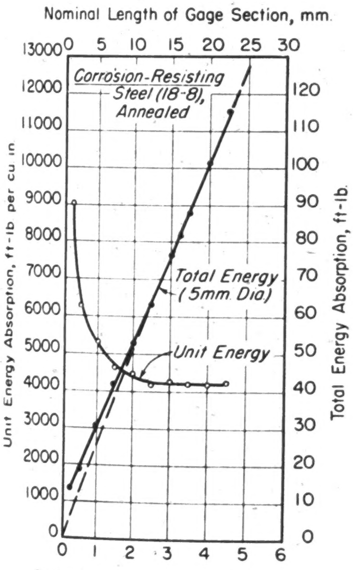


FIG. 6.—Effect of L/D Ratio on Unit Energy Absorption Values Obtained in Tension-Impact Tests of Corrosion-Resisting Steel (18–8), Annealed.

Tests conducted at room temperature. Striking velocity, 16.5 ft. per sec.

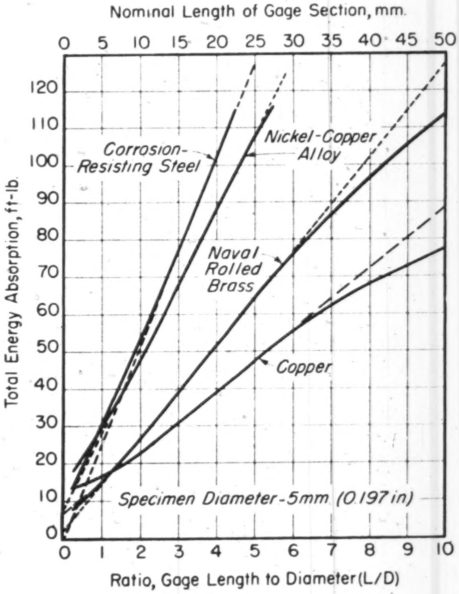


FIG. 8.—Effect of L/D Ratio on Total Energy Absorption Values Obtained in Tension Impact.

Tests conducted at room temperature. Striking velocity, 16.5 ft. per sec.

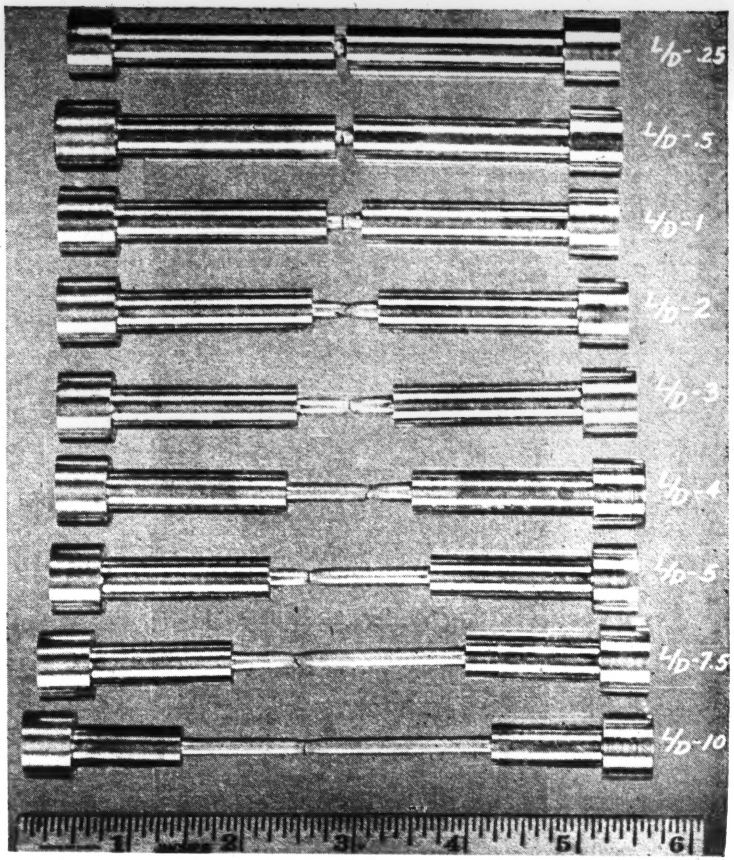

L/D-.25
L/D-.5
L/D-1
L/D-2
L/D-3
L/D-4
L/D-5
L/D-7.5
L/D-10
1
2
3
4
5
6

FIG. 10.—Fractured Tension-Impact Specimens of Naval Rolled Brass.

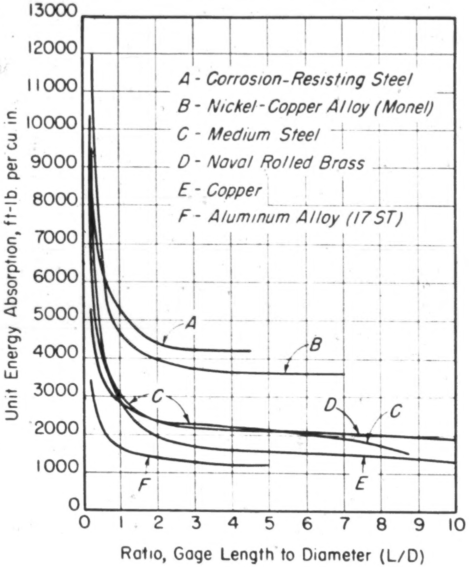


FIG. 9.—Effect of L/D Ratio on Unit Energy Absorption Values Obtained in Tension Impact.

Tests conducted at room temperature. Striking velocity, 16.5 ft. per sec. except in the case of B, where the striking velocity was 27.8 ft. per sec.

approximately $2\frac{1}{2}$ to 3 and extends to an L/D ratio of approximately 5 to $5\frac{1}{2}$. It is to be further noted that the total energy curves are substantially straight lines in this region. In the case of nickel-copper alloy, corrosion-resisting steel, and the aluminum alloy, it was not possible to test specimens with L/D ratios larger than those indicated because of the limited capacity of the testing machine used (120 ft-lb.).

The relative tensile impact resistance of the various materials may be compared by the relative slopes of the straight portions of the total energy curves (for specimens of the same diameter) indicated in Fig. 8, that is, the steeper the slope, the greater is the tensile impact resistance of the material. The height of the horizontal portion (say between L/D of 3 to 5) of the unit energy absorption curves shown in Fig. 9 may also be used to compare relative tensile impact resistance.

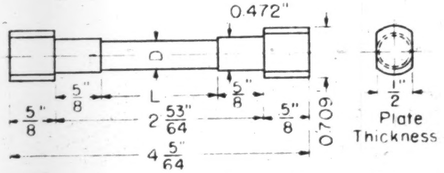
Mann (4) plotted energy per unit volume against the notch length L which yields a curve of the same general shape as those shown in Fig. 9, since, for a given diameter, the L/D ratio is directly proportional to the notch length. Mann's curve reveals the presence of three distinct zones (as was substantiated by the authors): for notch lengths of less than approximately 0.15 in. a zone of energy concentration increasing to extremely high values as the notch length is decreased; for notch lengths between approximately 0.15 and 0.60 to 0.80 in., a zone of constant unit energy absorption; and for longer notch lengths, a zone of gradual decrease in energy values. Mann used specimen diameters of 0.252, 0.357, and 0.437 in. For a notch length of 0.15 in., the corresponding L/D ratios would be 0.60, 0.42, and 0.34, respectively; and for a notch length of 0.80 in., the corresponding L/D ratios would be 3.18, 2.24, and 1.83, respectively. In this respect, the authors' results do not agree with those of Mann (4), since it was found (Fig. 9) that specimens with L/D ratios of less than approximately 2 yielded energy values falling in the zone of energy concentration. Mann apparently indicates that the three zones are defined by notch length alone, regardless of the diameter, but the authors' work does not bear this out, as may be seen by reference to Table I. The L/D ratio fully defines the geometry of the section under test whereas the length of the notch L only defines one dimension.

It appeared to the authors that a specimen with an L/D ratio of 4, which falls in the zone of constant unit energy absorption, would be suitable for use in connection with the contemplated work on the medium steel ship plate. The selection of an L/D ratio of 4 has the added advantage that the percentage of elongation obtained in tension impact may be compared to that obtained on standard 0.505-in. diameter by 2-in. gage tension specimens, in which the L/D ratio is also equal to 4.

Having decided on an L/D ratio of 4, the next step was to select specific values of D and L. In the case of the plate material under investigation, it was felt that D should be made as large as possible in order to provide for a test section which is fully representative of the plate thickness of approximately $\frac{1}{2}$ in. It was tentatively proposed to prepare specimens with $D = 10$ mm. (0.394 in.) and $L = 40$ mm. (1.576 in.) for test in an 1800 ft-lb. Amsler impact testing machine at a striking velocity of 27.8 ft. per second corresponding to the maximum available kinetic energy of the pendulum, which velocity is well below the transition velocity (6). In view of the fact that testing machines of this capacity are not in common use, it was considered desirable also to prepare smaller specimens, geometrically similar to the 10 by 40-mm. specimen, for test in the 120 ft-lb. Amsler machine. A specimen with a gage diameter *D* of 5 mm. (0.197 in.) and a gage length *L* of 20 mm. (0.788 in.) (L/D = 4) was selected for this purpose. It is realized that the smaller specimen may not be fully adequate since the gage diameter is not representative of the ½-in. plate thickness. However, it was felt that a specification requirement (if such be found feasible) should not be based on a specimen size which is not capable of being universally tested. The results of tests on specimens of both sizes, when completed, will serve to indicate whether variations in the homogeneity of the plate material permit any satisfactory correlation between specimens of appreciably different sizes. If such correlation exists, the incorporation of a specification requirement can be based on a specimen size which the average laboratory is in a position to test.

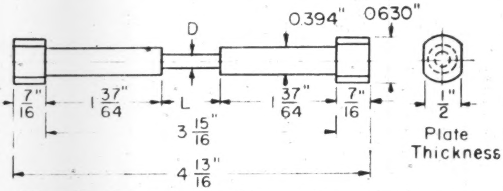
In order to determine whether specimens of the above sizes could be ruptured within the available capacities of the testing machines, specimens of both sizes were prepared from ½-in. medium steel plate (as-rolled) and tested with results as indicated in Table VIII. Results of tests on specimens with L/D ratios of 5 have also been included for comparison purposes. The gage volume of the smallest specimen (5 by 20 mm.) was arbitrarily called unity and the gage volumes of all other specimens have been compared to this by a suitable ratio. It is to be noted that good correlation is shown between the results obtained on the small and large specimens. For example, the 10 by 40-mm. specimen took an average of 485 ft-lb. which is nearly equal to 8 times that required to rupture the 5 by 20 mm. specimen (60.8 ft-lb.). Similarly, the 10 by 50-mm. specimen has a gage volume 10 times that of the 5 by 20-mm. specimen, and would therefore be expected to break at approximately 608 ft-lb.; the actual average value obtained was 609 ft-lb. Furthermore, when reduced to a common denominator, it is to be noted that, regardless of size, all specimens showed unit energy absorption values which

TABLE VIII.—TENSION IMPACT SPECIMENS AND RESULTS OF TESTS ON ½-IN. MEDIUM STEEL PLATE, AS-ROLLED. TESTS MADE AT ROOM TEMPERATURE.



1800 FT-LB. AMSLER IMPACT TESTING MACHINE; STRIKING VELOCITY, 27.8 FT. PER SEC.

D, mm.	L, mm.	L/D	Gage Volume, cu. in.	Volume Ratio[a]	Total Energy, ft-lb.	Unit Absorption, ft-lb. per cu. in.	Elongation, per cent[b]	Reduction of Area, per cent
10	50	5	0.24	10	600	2500	38.0	65.3
10	50	5	0.24	10	618	2570	40.0	65.0
				Avg.	609	2535	39.0	65.2
10	40	4	0.192	8	480	2500	38.1	64.5
10	40	4	0.192	8	490	2550	39.4	66.4
				Avg.	485	2525	38.8	65.5



120 FT-LB. AMSLER IMPACT TESTING MACHINE; STRIKING VELOCITY, 16.5 FT. PER SEC.

6	30	5	0.0518	2.16	No break			
6	30	5	0.0518	2.16	No break[c]			
6	24	4	0.0415	1.73	105.0	2530	36.4	63.8
6	24	4	0.0415	1.73	104.5	2520	35.4	62.0
				Avg.	104.8	2525	35.9	62.9
5	20	4	0.024	1.00	59.0	2460	35.0	63.0
5	20	4	0.024	1.00	62.5	2600	38.8	64.1
				Avg.	60.8	2530	36.9	63.6

[a] Ratio of gage volume to that of 5 by 20-mm. specimen.
[b] Per cent elongation in the respective gage length.
[c] Using the volume ratio, it is estimated that these specimens would have required approximately 2.16 x 60.8 = 131.5 ft-lb. which is beyond the available capacity of 120 ft-lb.

agree closely from one specimen to the next. These tests, of course, have all been based on a specimen design which lies in the zone of constant unit energy absorption.

To investigate further the effect of size, a series of specimens of the form shown at the top of Table VIII was prepared from the nickel-copper alloy (monel) stock, with a gage diameter of 0.300 in. and various *L/D* ratios. These specimens were tested at room temperature in the 1800 ft-lb. Amsler machine, the results of which are given in Table IX. A comparison of the figures obtained on

TABLE IX.—RESULTS OF TENSION-IMPACT TESTS OF NICKEL-COPPER ALLOY (MONEL), ANNEALED AT 1500 F.

Tests conducted at room temperature on specimens of the form shown in sketch at top of Table VIII. Striking velocity, 27.8 ft. per sec.

Nominal *D*, in.	Nominal *L*, in.	*L/D*	Total Energy, ft-lb.	Gage Volume, cu. in.	Unit Energy, ft-lb. per cu. in.
0.300	0.075	0.25	60	0.0054	11 100
0.300	0.150	0.5	75	0.0107	7 010
0.300	0.215	0.75	80	0.0153	5 230
0.300	0.30	1.0	90	0.0212	4 250
0.300	0.45	1.5	130	0.0321	4 050
0.300	0.60	2.0	180	0.0431	4 180
0.300	0.75	2.5	210	0.0534	3 930
0.300	0.90	3.0	245	0.0641	3 820
0.300	1.05	3.5	270	0.0746	3 620
0.300	1.20	4.0	320	0.0850	3 770
0.300	1.35	4.5	355	0.0958	3 710
0.300	1.50	5.0	405	0.1060	3 820
0.300	1.65	5.5	420	0.1164	3 610
0.300	1.80	6.0	455	0.1276	3 570
0.300	1.95	6.5	500	0.1377	3 630
0.300	2.10	7.0	540	0.1489	3 630

TABLE X.—COMPARATIVE RESULTS OF TENSION-IMPACT TESTS OF LARGE AND SMALL SIZE SPECIMENS OF NICKEL-COPPER ALLOY (MONEL) ANNEALED AT 1500 F.

Tests conducted at room temperature.

L/D	Gage Volume, cu. in.		Volume Ratio[a]	Total Energy, ft-lb.			Unit Energy, ft-lb. per cu. in.	
	5-mm. Diameter (0.197 in.)	0.300 in. Diameter		5-mm. Diameter (0.197 in.)	0.300 in. Diameter		5-mm. Diameter (0.197 in.)	0.300 in. Diameter
					Actual	Calculated		
0.25	0.0015	0.0054	3.60	17.9	60	64.4	11 930	11 100
0.5	0.0030	0.0107	3.57	21.75	75	77.6	7 250	7 010
1.0	0.0059	0.0212	3.59	29.15	90	104.6	4 900	4 250
1.5	0.0091	0.0321	3.53	37.5	130	132.4	4 120	4 050
2.0	0.0119	0.0431	3.62	47.5	180	172.0	3 990	4 180
2.5	0.0153	0.0534	3.49	58.5	210	204.2	3 820	3 930
3.0	0.0179	0.0641	3.58	65.3	245	233.8	3 645	3 820
3.5	0.0207	0.0746	3.60	77.5	270	279.0	3 740	3 620
4.0	0.0238	0.0850	3.57	86.5	320	308.8	3 640	3 770
4.5	0.0269	0.0958	3.56	99.8	355	355.3	3 710	3 710
5.0	0.0298	0.1060	3.56	107.4	405	382.3	3 605	3 820
5.5	0.0329	0.1164	3.54	116.3	420	411.7	3 540	3 610

[a] Ratio of gage volume of 0.300-in. diameter specimen to gage volume of 5-mm. (0.197-in.) diameter specimen.

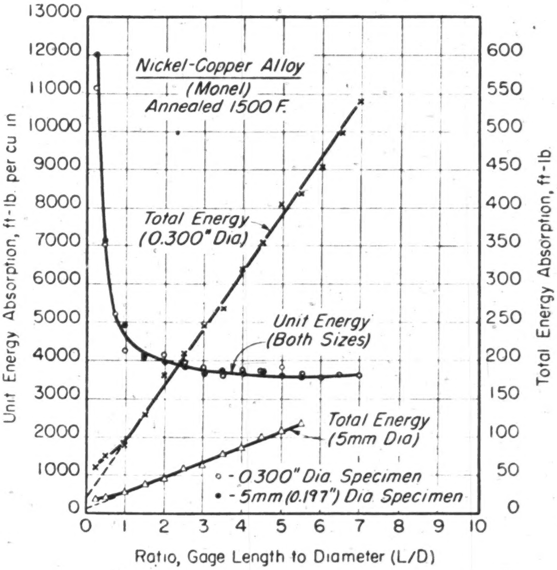


FIG. 11.—Effect of *L/D* Ratio on Energy Absorption Values Obtained in Tension-Impact Tests of Nickel-Copper Alloy (Monel), Annealed at 1500 F.

Tests conducted at room temperature. Striking velocity, 27.8 ft. per sec. for 0.300-in. diameter specimens and 16.5 ft. per sec. for 5-mm (0.197 in.) diameter specimens.

the large and small size specimens of this material has been made in Table X. The calculated total energy values for the 0.300-in diameter specimens were obtained by multiplying the values for the 5-mm. diameter specimens by the volume ratio. It is to be noted that good correlation (within the limits of experimental error) is shown between the calculated and the actual total energy values, particularly in the zone of approximately constant unit energy absorption. The unit energy absorption values also show close agreement in this zone. In Fig. 11, where the results of tests on specimens of both sizes have been plotted, it is to be observed that points representing the unit energy values lie close to a common unit energy absorption curve.

CONCLUSIONS

To evaluate properly the dynamic properties of metals in tension impact, as determined by tests conducted in a conventional pendulum type impact testing machine, and in order to express the results on a common basis, the following factors should be observed:

(*a*) The gage section of the test specimen should be proportioned with an L/D ratio of between 3 and 5, preferably 4. In this connection, the diameter of the gage section should not be made too large in proportion to the diameter of the shoulders in order to avoid plastic deformation of the shoulders.

(*b*) The results of tests should be expressed in terms of unit energy absorption, that is, the number of foot-pounds of energy absorbed per cubic inch of metal contained in the gage section. The use of this unit permits comparison of tests made on specimens of appreciably different size and also eliminates discrepancies resulting from dimensional variations in machining specimens of the same nominal gage diameter and length.

Acknowledgment:

The authors wish to acknowledge the assistance received from members of the Material Laboratory of the New York Naval Shipyard who participated in the conduct of the investigation and also to express their appreciation to the Bureau of Ships, Navy Department, for permission to publish the results thereof.

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(3) P. G. Jones, "On the Transition from a Ductile to a Brittle Type of Fracture in Several Low-Alloy Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 547 (1943).

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(5) C. F. Catlin and W. A. Mudge, "Compressive, Impact, and Other Mechanical Properties of Nickel and Some of Its High-Strength, Corrosion-Resisting Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 269 (1938).

(6) H. C. Mann, "High-Velocity Tension-Impact Tests," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, p. 85 (1936).

DISCUSSION

MR. T. J. DOLAN[1] (*presented in written form*).—The authors are to be congratulated for presenting some very interesting and rather extensive data and for their emphasis of the need for standardization of a tension-impact test specimen. The wide variety of impact test data reported by various investigators makes it difficult if not impossible to interpret or correlate the results in terms of basic characteristics of the materials involved.

The writer would like to comment further on some of the factors mentioned on page 1183 of the paper, as influencing the unit energy absorption as the L/D ratio is increased. If one visualizes the energy absorbed by each small element of the specimen to be approximated by the area under a *true* stress-strain diagram for the element up to the strain it receives during rupture of the specimen, an approximate energy distribution diagram might be plotted as shown in the accompanying Fig. 13(*a*). Due to necking of the specimen, smaller areas of metal are associated with the higher rates of energy absorption near the fracture than the areas of the zones of restricted energy absorption near the ends. For rough comparative purposes it is therefore more convenient to visualize the original specimen as divided into three different zones as shown in Fig. 13(*d*). The relative averages of energy absorbed per unit volume in each zone (and based on the full diameter D) might then be represented by some approximate numerical values E_1, E_2, E_3.

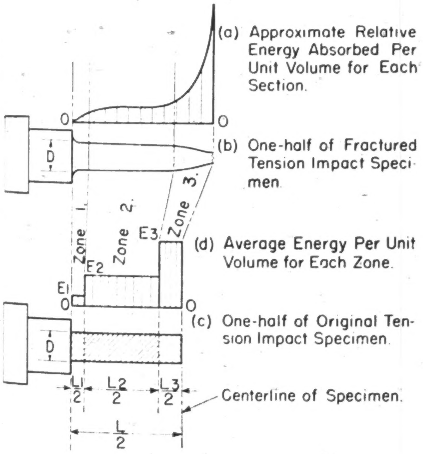
(a) Approximate Relative Energy Absorbed Per Unit Volume for Each Section.
O
O
D
(b) One-half of Fractured Tension Impact Specimen.
Zone 1
Zone 2
Zone 3
E3
E2
E1
O
O
(d) Average Energy Per Unit Volume for Each Zone.
D
(c) One-half of Original Tension Impact Specimen.
L1/2
L2/2
L3/2
L/2
Centerline of Specimen.

FIG. 13.—Energy Absorption by One Half of a Tension-Impact Specimen.

$$\text{Energy per unit volume} = \frac{E_1L_1 + E_3L_3 + E_2L_2}{L}$$

Based on this concept, the energy absorbed in fracturing the complete specimen would be:

Total Energy

$$= [E_1 L_1 + E_2 L_2 + E_3 L_3] \frac{\pi D^2}{4} \quad \text{..(1)}$$

and, dividing by $\dfrac{\pi D^2 L}{4}$:

The Average Energy per Unit Volume

$$= \frac{E_1 L_1 + E_3 L_3}{L} + \frac{E_2 L_2}{L} \quad \text{..(2)}$$

and

The total gage length, $L = L_1 + L_2 + L_3$..(3)

---

[1] Research Professor of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

In general, the effective lengths L_1 and L_3 are probably functions of the kind of material and temperature of testing. The length L_2 (of fairly uniform elongation) is nonexistent for short gage lengths but will increase as the specimen is lengthened beyond the value for which $L = L_1 + L_3$. Thus the energy per unit volume obtained in a tension-impact test is a weighted average of these actions occurring in portions of the specimens whose lengths are functions of specimen size, temperature, and type of material.

As the length of specimen is greatly increased (beyond that at which interference between zones 1 and 3 occurs) the total contributions of zones 1 and 3 probably do not change materially, and for long specimens the total energy absorbed tends to approach more and more closely that contributed by the large volume of material in zone 2. In tension-impact tests of a cold-rolled steel by Clark[2] the full reduction of area occurred only for specimens with $L = 2D$ or greater, but no appreciable change was noted in reduction of area in longer specimens. This might be interpreted as indicating that interference of zones 1 and 3 decreased the reduction of area in very short specimens, but that variations in the length of zone 2 introduced by testing specimens longer than $L = 2D$ did not cause material alterations in the maximum strains developed in zone 3. For other metals the gage length at which this transition occurs (and a zone 2 is introduced) might be somewhat different depending on the elongation and reduction of area characteristics of the metal.

If the unit energy value E_2 is approximately equal to the average energy per unit volume of the combined zones 1 and 2, changes of the gage length should lead to test data (of the type shown in the authors' Fig. 6) having a horizontal tangent indicating constant energy absorption per unit volume as the ratio L/D is increased to very large values. On the other hand, if E_2 is less than the average in zones 1 and 3, the test data would indicate a curve of gradually decreasing curvature with no horizontal portion as L/D is increased, but tending to approach a horizontal asymptote for very long gage lengths. This appears to be the case in the majority of the authors' curves (such as Figs. 1, 3, and 4).

Based on these considerations it does not seem reasonable to suspect an abrupt falling off of energy absorption as the gage length is increased beyond eight or ten diameters, nor in general may we presume that all materials would exhibit a constant energy absorption for specimens with L/D ratios of 3 to 5. It is suggested that the one set of low values of energy absorption shown for $L/D = 9$ in the authors' Fig. 1 might be attributable to fortuitous conditions during the test. In general, less drop in energy absorption per unit volume should be expected as the L/D ratio is increased from 8 to 10 or larger, than the drop in energy absorption as the L/D ratio is increased from 3 to 5 (the latter being the range of gage length recommended by the authors).

The writer has in various groups of tests used specimens identical in design with an L/D ratio of 10 in the hopes of minimizing some of the variables present. A gage length of such a value is of advantage in averaging more material when one is restricted to a small diameter specimen by the limited capacity of the testing machine. However, as indicated above, the energy to rupture a long specimen is predominantly that required to cause the large amount of uniform elongation, whereas for relatively short specimens the energy measured is mainly that developed in the concentrated zone of necking. Hence it is theoretically possible that for specimens with $L/D = 3$ a metal A (which necks down appreciably) might have a higher tensile impact resistance per unit volume than metal B, while for specimens with $L/D = 10$, metal B may show higher unit energy absorption due to its greater tendency for uniform elongation. This is perhaps all the more reason for standardization of tension-impact specimens for comparative purposes.

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[2] D. S. Clark, "The Influence of Impact Velocity on the Tensile Characteristics of Some Aircraft Metals and Alloys, Nat. Advisory Comm. Aeronautics, Tech. Note No. 868, October, 1942.

The first question to consider, however, in standardizing on a tension-impact specimen is: *what property of the material do we wish to emphasize or evaluate?* If the quality we are interested in is associated with the ability of the metal to absorb energy locally in regions of stress concentration, this property might best be measured by the energy values obtained from short gage length specimens, and the energy absorbed in necking may predominate. On the other hand, if it is important to evaluate the ability of the metal to absorb energy in larger areas of a member subjected to fairly constant stress, a long gage length specimen is probably more representative in exhibiting the property desired. Under the embrittling conditions encountered in certain tests at low temperatures (for which the reduction of area becomes small or negligible), a relatively long specimen would be of advantage in attempting to measure the small differences of energy absorption of various metals that might be due to the small general elongation, whereas a short specimen would accentuate mainly the energy required to produce a brittle fracture in a region of stress concentration and not accompanied by appreciable general yielding. Perhaps what is needed is to standardize on two types of tension-impact specimen for the evaluation of two slightly different properties of the material.

In several groups of tests, the writer has found that the unit energy absorption in a tension-impact test was appreciably greater than the toughness of the metal as measured by the area under a static stress-strain diagram. We have attributed this difference mainly to energy loss on collision of the striking faces and to energy loss by transference to ground through the impact machine. The tests of Clark referred to above also seem to indicate this same tendency toward greater energy absorption in an impact test than that of a static test. It would be interesting to know whether the authors have also obtained higher values from tension-impact tests than the toughness as measured from static tension test data.

MR. COMFORT A. ADAMS.[3]—What is the speed of impact?

MR. E. A. IMBEMBO (*author*).—The striking velocity in the case of the small-size specimens was 16½ ft. per sec. On the larger specimens, which were broken in the 1800 ft-lb. Amsler testing machine, the striking velocity was 27.8 ft. per second, so that the striking velocity for the large and small-size specimens was not the same. Nevertheless, we feel that over that range of striking velocity, the energy absorption values would not be materially different, since these velocities are considerably below the critical velocities for the materials tested.

MR. G. A. OLSEN.[4]—I was very much interested in what Mr. Imbembo had to say concerning the impact tension specimen. As to distinguishing among various metals, that might be a very good thing. I was wondering, however, just how close the foot pound per cubic inch energy value would be to the ideal value. In other words, how close would it be to the maximum value that every cubic inch might possibly take?

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[3] Consulting Engineer, The Budd Co., Philadelphia, Pa.
[4] Bakelite Corp., Bloomfield, N. J.

MR. IMBEMBO.—I think Mr. Dolan has attempted to integrate the distribution of energy along the length of the specimen to divide it up into three zones. It is perfectly true that each increment of the specimen gage section does not absorb the same amount of energy because the deformation of the material varies from the necked-down portion to the end of the gage section.

What we have given here is an integrated value of the energy absorption throughout the gage length of the specimen. I do not know whether that answers what you have in mind. Perhaps I do not understand the question properly.

MR. OLSEN.—The striking edge, or whatever the hammer is, is stressed at the time of impact and undoubtedly the energy which went into stressing it, is a portion of the total energy. The shoulder itself was stressed, a certain amount of energy going into that. What per cent of the total energy do you suppose went into the specimen?

MR. IMBEMBO.—There is a certain amount of energy included in the actual energy value that is read on the scale of the machine which is taken up within the machine itself. Throwing off the broken portion of the specimen, for example, also involves an energy absorption, for which we have not made any correction, since it is a quantity difficult to evaluate, at any rate, this quantity is small in comparison to the total energy absorption.

MR. N. A. KAHN (*author*).—I want to add a remark in connection with the discussion by Mr. Olsen. I have observed in notched-bar, cantilever-type, impact tests on plastics that energy values as low as 1 ft-lb. are occasionally obtained. When the discarded portion is replaced over the portion remaining in the machine and the pendulum swung a second time from the original position, the scale would indicate as much as $\frac{3}{4}$ ft-lb. as the energy absorbed in simply throwing out the fractured portion into the air. In connection with steel test specimens, fortunately, we are not confronted with this difficulty.

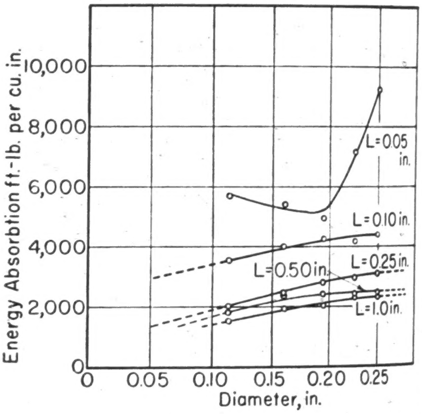

FIG. 14.—Effect of Diameter on Unit Energy Absorption Values in Tension-Impact Tests of Medium Steel Rivet Rod.

MR. P. G. JONES[5] (*by letter*).—The authors have presented some very interesting results of their study of the geometry of the impact test specimen. Since it is sometimes necessary to compare results on specimens of different sizes because of the requirements of the testing machine or because of size of stock, the writer is in agreement with the authors that there is need for a method of correlating these results.

In order to express the energy results on a common basis of energy per unit volume, the authors propose specifying a limit to the ratio of L/D of between 3 and 5. On the basis of the data presented in the paper, it would appear that this criterion would be satisfactory. However, the writer questions the constancy of energy per unit volume for a constant ratio of L/D if extreme values of L and D are used. The authors' data on the medium steel rivet rod is shown in Fig. 14 of this discussion in which the energy absorbed per unit volume is plotted against the diameter of the specimen for specimens having constant length. From these curves and from curves of unit energy plotted against L for specimens of constant diameter (not presented with this discussion), one may obtain simultaneous values of L and D which would give constant energy absorption. For example, in order for a specimen to absorb 2000 ft-lb. per cubic inch of energy it may be seen from the curves that if the length is 0.25 in., the diameter should be about 0.105 in.; for a length of 0.50 in., the diameter should be about 0.125 in.; and for a length of 1.0 in., the diameter should be about 0.175 in. These data have been obtained for several values of energy and the results are shown in the accompanying Fig. 15 in which L is plotted against D. One would expect the constant energy curves to pass through the origin. However, this is not the case, thus indicating that a constant value of L/D will not necessarily give a constant value of energy per unit volume.

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[5] Assistant Professor in Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

The authors' data given in Table VIII for ½-in. medium steel plate seem to refute this argument. However, it should be noticed that portions of some of the curves shown in Fig. 15 when extrapolated will very nearly pass through the origin. In other words, the ratio of L/D may be satisfactory provided the variation in L and in D is not too great. For that matter a constant length could be specified with limits placed on the diameter or a constant diameter could be specified with limits placed on the length and one would expect the energy per unit volume to check very closely. A ratio of $\frac{L}{D^2}$ or a ratio of length to area might be a better quantity to specify than the ratio of L/D. It will be observed that if in Fig. 15 of this discussion the length were plotted against D^2 instead of D the curves would more nearly pass through the origin.

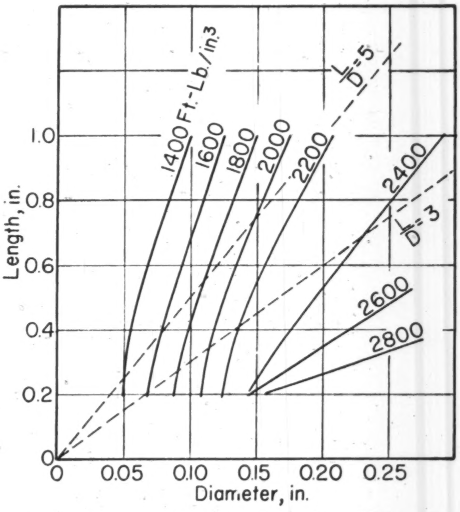

FIG. 15.—Relation Between Length and Diameter of Tension-Impact Specimens to Give Constant Energy Absorption.

The authors point out that specimens with very short notch lengths are capable of absorbing relatively large amounts of energy as indicated by the unit energy values in Table I, but that the total energy values are small because of notch effect. It might be added here that the high stress concentration at the root of the notch and the triaxial state of stress which accompanies a short notch length may cause extremely low energy per unit volume in addition to low total energy values when the tests are made at low temperature or at very high rates of strain.

MESSRS. N. A. KAHN AND E. A. IMBEMBO (*authors' closure by letter.*)—The authors wish to thank all those who contributed their time and effort to the discussion of their work, and are particularly appreciative of the interesting and extensive written discussions prepared by Messrs. Dolan and Jones.

The discussion by Mr. Dolan suggests that one must ascertain what property of the material is to be evaluated before standardizing the tension-impact specimen. We are in general agreement with him that when one is interested in the ability of a metal to absorb energy locally in regions of stress concentration, an especially designed test specimen of short gage length is required. However, the authors' paper deals with the design of a specimen for the evaluation of the ability of a metal to absorb energy in larger areas subjected to fairly constant stress, in which connection we found that an *L/D* ratio of approximately 3 to 5 exhibited practically constant unit energy absorption for a representative variety of commercial metals, such as copper, Naval rolled brass, mild steel, monel, aluminum alloy, and austenitic corrosion-resistant steel.

The authors' observations on all of these materials indicate a definite dropping off in unit energy absorption for large values of *L/D*. The case cited by Mr. Dolan involving austenitic corrosion-resistant steel (Fig. 6), as an example of a material having a horizontal tangent indicating constant energy absorption per unit volume as the *L/D* ratio is increased to very large values, appears to be misleading in that, due to limitation in the capacity of the testing machine, an *L/D* ratio of 4.5 was the maximum tested and reported.

In connection with the question raised by Mr. Dolan in the last paragraph of his discussion, the authors did not undertake an evaluation of the toughness of the various materials as measured from static tension test data and, hence, are not in a position to indicate directly whether the static tension-energy values would be less than the tension-impact energy values. However, one of the authors had occasion to consider this matter back in 1934.[6] At that time, a search of the literature indicated that the energy absorbed in the tension-impact test is generally greater than that absorbed in the static tension test.

Honda[7] showed that the work required for a sudden break is proportional to the volume of material subjected to stress and is not proportional to the cross-sectional area of the test specimen. If the deformation in the static test is the same as that in the dynamic test, the energy absorbed in each case should be the same since the work is chiefly determined by the energy of deformation. In the case of tension tests, it has, however, been observed[7, 8] that the energy absorbed in the impact test is generally greater than that absorbed in the slow tension test. Honda[7] states that this difference is due to the difference in deformation in these two tests. He goes on to state that although the total elongation may be the same in both tests, the local elongation may be greater in the dynamic test so that the energy absorbed would be greater in the latter test. Honda's results[7] show that the work of rupture for carbon steels in the dynamic test is greater than that in the static test, as indicated in the following table:

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[6] E. A. Imbembo, "Effect of Low Temperature on the Tensile Impact Resistance of Some Welded Joints," Thesis submitted in partial fulfillment of the requirements for the Degree of Master of Mechanical Engineering at the Polytechnic Institute of Brooklyn, June 1, 1934.

[7] Kotaro Honda, "A Comparison of Static and Dynamic Tensile and Notched Bar Tests," *Journal*, Inst. Metals, Vol. 36, No. 2, pp. 27-37 (1926).

[8] B. Blount, W. G. Kirkaldy, and H. R. Sankey, "Comparison of Tensile, Impact Tensile, and Repeated Bending Methods of Testing Steel," *Proceedings*, Institution Mechanical Engrs. (London), No. 2, pp. 715-772 (1910).

Carbon Content, per cent	Ratio, Dynamic to Static
0.1	1.28
0.3	1.48
0.5	1.41
0.7	1.34
0.9	1.32

Blount, Kirkaldy, and Sankey,[8] as a result of tests on unnotched tension specimens of 13 carbon steels, found that the ratio of the work absorbed in the impact test to that absorbed in the slow tension test varied from 1.31 to 1.75, the average being 1.58. They also found that, in every case, the elongation and reduction of area were greater in specimens broken under impact than in those broken in slow tension. Philpot[9] assumes, as does Mr. Dolan, that the greater energy value is due to transformation of part of the kinetic energy of the pendulum into vibrations of the machine. However, since there is some evidence to indicate that ductility is greater in tension impact than in static tension, it does not appear probable that the difference in energy values can be entirely attributed to energy loss in the testing machine.

Jones appears to agree that a limit of 3 to 5 for the L/D ratio is a satisfactory criterion for the tension-impact specimen. In connection with Fig. 14, in which he has plotted energy per unit volume against diameter of the specimen for specimens of constant length, he suggests that such a plot might be utilized to determine the dimensions of a specimen for constant energy absorption. It is interesting to note that the example quoted involves L/D ratios of 2.4, 4.0, and 5.7, respectively, which are all within the range of practically constant unit energy absorption. The authors do not quite grasp the significance of Fig. 15 and believe that a plot of length against D^2 might have provided more pertinent information, as suggested by Jones.

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[9] M. P. Philpot, "Some Experiments on Notched Bars," *Journal*, Soc. Automotive Engrs., Vol. 3, No. 6, pp. 347-357. December, 1918.

SYMPOSIUM ON FREEZING-AND-THAWING TESTS OF CONCRETE

CONSIDERATIONS INVOLVED IN THE MAKING OF FREEZING-AND-THAWING TESTS ON CONCRETE*

By M. O. Withey[1]

Synopsis

The paper emphasizes the importance of a knowledge of the weathering resistance of concrete. The objective of the test and the desired acceleration of the rate of disintegration without modification in the process of disintegration is stressed. Factors influential in developing freezing-and-thawing tests and the types of tests commonly used are considered. The more important details of procedure for testing concrete containing aggregate of uncertain quality, the principal indices of damage, and the selection of the end point are discussed. A brief consideration is also made of the significance of freezing-and-thawing tests and the further research which is needed to provide proper standards for procedure.

The numerous alternations of freezing and thawing (often 25 to 40 per annum) in the climate of the temperate zones cause deterioration in all porous rock and masonry structures. Concrete and mortar are particularly susceptible to the weathering influences of nature when subjected to severe exposure. Hence the designer, builder, and owner of structures should know the relative durabilities of different grades of such materials in order to appraise their value in construction. Desire for such information has led to the use of numerous freezing-and-thawing and other types of accelerated weathering tests. In these tests the objective has been to secure, in a relatively short period of time, an index of the length of service which may be expected of the material under test when subjected to a given type of exposure. Of the accelerated tests thus far tried, the freezing-and-thawing test, which approaches conditions in nature, has been the most satisfactory.

It will be the purpose of this paper to enumerate some of the factors to be considered in developing a satisfactory test, to indicate the types of test used, to detail a test procedure which appears to have much merit in testing concretes made from aggregates of questionable quality, to consider indices of damage during tests, methods of determining the end point of the test, the significance of such accelerated tests, and to point out some needed research.

Factors Influential in Developing a Test

Since the value of the index of durability determined by the data from a freezing-and-thawing test is dependent upon the correlation which exists between that index and the action of nature, the test should be so devised that it will correspond in its action to that of nature. The only important divergence should be that the test sets up a much more rapid *rate* of disintegration in test specimens without modifying the *process* of disintegration accomplished much more slowly by nature.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] Professor of Mechanics, The University of Wisconsin, Madison, Wis.

It seems apparent from studies made by Kreuger (**1**),[2] Powers (**2**), and others that the deteriorating influence of freezing and thawing is intimately related to: (1) the permeability of the concrete; (2) the degree to which the concrete is saturated with freezable water; and (3) the amount of freezable water in the concrete.

These factors will be materially influenced by the water-cement ratio of the paste (**3**), the character of the aggregate (**4**), the consistency of the concrete (**5**, **6**), the curing conditions (**6**), and the proportion of entrained air in the set concrete (**7**). Factors 2 and 3 will also be affected by the type of exposure. In a rigorous climate, concrete in the exposed face of an exterior wall above grade will suffer less deterioration than that in a parapet wall or similar concrete in contact with a wet soil or partly immersed in water. Test data indicate (**8**) that the rate of disintegration of concrete increases with the rate that the temperature falls during freezing. Since the fall of atmospheric temperature in the vicinity of 32 F. rarely exceeds 3 F. per hour, it does not appear desirable to lower the temperature during a freezing test at too fast a rate. Until more determinative data are obtained, a limit of 10 F. per hour is suggested.

TYPES OF TEST COMMONLY USED

The three types of freezing-and-thawing tests commonly used are:

1. Freezing immersed in water and thawing submerged in water.
2. Freezing partly immersed in water and thawing partly or wholly immersed in water.
3. Freezing in air after saturating in water and thawing submerged in water.

In each of these types of test there have been a great many variables: the temperature of the specimen when placed in the freezer, the rate of freezing, the minimum temperature attained, the temperature of the water used in thawing, and the rate of thawing. In several laboratories operating under type 1, each small test beam is placed in a special form of rubber boot filled with water (**9**). The boots are immersed in a stirred solution of brine or alcohol maintained at a low temperature by means of cooling coils. In thawing, the boots are transferred to a water tank maintained at a fixed temperature above the freezing point. By this procedure very rapid rates of freezing may be provided without subjecting the specimens to the direct action of the brine or alcohol.

A committee on the durability of concrete sponsored by the Highway Research Board of the National Research Council reported (**9**) in 1944 on the results of a program of tests involving the cooperation of eight laboratories. In the rather extensive program pursued, all three of these methods were used by one or more of the cooperating laboratories in testing a concrete of 0.55 water-cement ratio, by weight, in which the quality of the coarse aggregate was made variable. The procedure of type 3, which is described in the following section, gave the most uniform indication of quality of the concretes tested. The detailed procedures for mixing, molding, curing, and methods of measuring the effects of freezing and thawing can well be applied in any one of the three freezing-and-thawing procedures listed.

PROCEDURE FOR TESTING CONCRETE CONTAINING AGGREGATE OF UNCERTAIN QUALITY

Materials:

All of the materials used in making the specimens for freezing-and-thawing tests of concrete must be correctly chosen and properly sampled if the subsequent tests are to be truly determinative of the durability of the concrete. If the quality of the aggregate is the questionable factor under test, then all other materials must be selected to avoid introducing other affecting variables. Cements with high alkali contents (10) or high tricalcium aluminate contents (8) should be avoided. The air-entraining characteristics of the cement (7) should be low. Cements should be thoroughly mixed and stored in tight metal containers during the tests.

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[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 1207.

Aggregates should be brought to the moisture content which they will contain in field use. The percentage of saturation and the variability in the water content of the coarse aggregate are factors of potent influence on the durability of concrete (4) and must be closely controlled. Aggregates should be graded within fixed limits in preparing all batches of concrete. The maximum diameter of coarse aggregate for concrete specimens with 3-in. minimum dimension should not exceed 1 in. It is well to weigh saturated coarse aggregate under water (9) and saturated surface wet sand in air. Allowance for water adhering to the surfaces of the aggregate can then be made in weighing the mixing water.

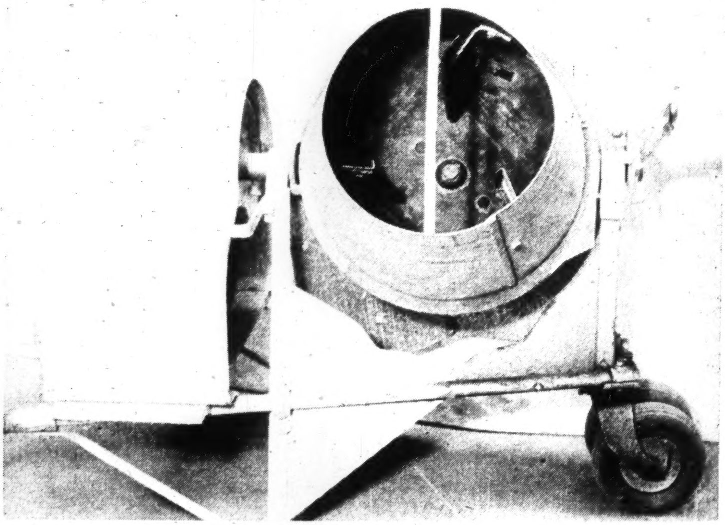


FIG. 1.—Mixer Used at the University of Wisconsin Laboratory.
Speed, 16 rpm. Capacity, 1⅓ cu. ft.

Mixing:

Machine mixing for a period of at least 5 min. is desirable to avoid variations in the air content of individual specimens. A suitable mixer is shown in Fig. 1. Both the materials and the air of the mixing room should be maintained at a uniform temperature, 70 ± 2 F., and the relative humidity of the mixing room should be between 50 and 60 per cent. The interior surfaces of the mixer should be maintained clean and brought to a uniform condition by wiping with a moist rag just before the batch is admitted. Each batch should be dumped onto a moistened pan and rendered uniform by remixing with a shovel. The slump or flow should then be determined. A slump of $2\frac{1}{2}$ to 3 in. is satisfactory for making small beams 3 by 4 by 16 in. in size or for small cylinders. Tests on the amount of entrained air in the fresh concrete should also be made in order to check the air content of each batch of concrete. This is especially important when treated cements are used.

Molding:

Metal molds, Fig. 2, consisting of cold-rolled steel plate sides fitted into slotted cold-rolled end and bottom plates are satisfactory. Joints should be made watertight by sealing with graphite grease, and interior surfaces should be given a thin coat of mineral oil before the molds are filled.

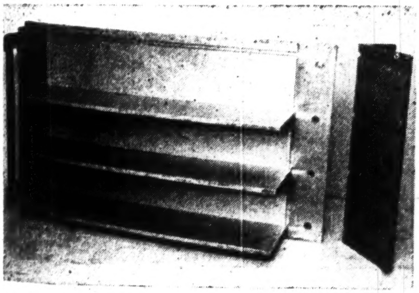


FIG. 2.—Molds of Cold-Rolled Steel Plate for 3 by 4 by 16-in. Concrete Beams.

Six 3 by 4 by 16-in. beams is a convenient number to mold at one time. In making small beams of the size mentioned, molding should be done in two layers. Each layer should be rodded 40 times with a $\frac{5}{8}$-in. bullet-nosed rod, spaded 25 strokes on each side and 10 strokes on each end with a 6-in. pointing trowel. In placing the second layer the molds should be filled $\frac{1}{8}$-in. above the top and rodded and spaded as before. The tops should be struck off level with a straight edge which should be advanced slowly along the beam and finished with three strokes of a wood float. Since it is believed that the amount of bleeding affects resistance to freezing and thawing, troweling of the surface should be minimized. If desired, beams of this size can also be equipped with plugs centered in the ends to permit measurements of expansion during the test.

Curing:

Filled molds should be covered with burlap held off the tops of the specimens and immediately placed in the moist closet. Molds and specimens should be protected from direct contact with the water spray. Specimens should remain in the moist closet for two days at a temperature of 70 ± 2 F. and a humidity of 100 per cent. The molds should then be removed and the specimens immediately weighed to the nearest gram, in air and then under water. This procedure provides data from which the air entrained in the set concrete may be estimated. Specimens should then be immersed in water at 70 ± 2 F. for 26 days. Proponents of a period of air curing of a week or 10 days introduced prior to the beginning of the freezing-and-thawing cycles claim that the quality of the concrete is thus rendered more uniform. However, such procedure will also materially lengthen the time required to break down the specimens in the freezing-and-thawing test.

A Suggested Procedure for Freezing and Thawing:

When the specimens become 28 days old they should be cooled for 5 to 7 hr. in water at 40 F. Specimens should then be weighed in air and in water at 40 F. Immediately thereafter specimens should be subjected to tests for the dynamic modulus of elasticity, E_d, taking care to maintain the temperature of the specimens as close to 40 F. as possible. In the dynamic modulus tests, rectangular beams may be subjected to vibration in each of the principal longitudinal planes and calculations can be made, if desired, for both of these determinations.

Figure 3 shows the apparatus for determining E_d. The beam specimen, B8-2, is supported at its nodal points at 22.4 per cent of its length from each end on three steel balls. It is vibrated laterally by a horizontal piston attached to the cone of a loud speaker housed in the heavy metal box *A*. The minute deflections of the right end of the beam actuate the crystal pick-up *B* and are indicated on the milliammeter at *C*. The operator is enabled to vary the frequency of the cycles of vibration imposed on the beam by turning the dial. Resonance is denoted by the maximum swing of the pointer.

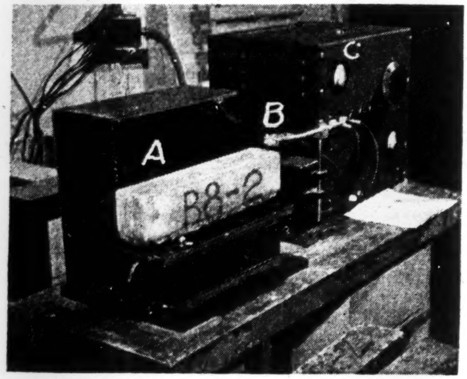


FIG. 3.—Apparatus for Determining the Dynamic Modulus of Elasticity of Small Concrete Beams.

The dynamic modulus of elasticity is calculated from

$$E_d = \frac{713.9pL^4f^2T}{m^4d^2}$$

where:

p = specific weight in pounds per cubic foot,
L = length of specimen in inches,
f = natural frequency in kilocycles,
d = depth of beam in plane of vibration, and
T = correction factor dependent on $\frac{d}{L}$ and Poisson's ratio.

For 3 by 4 by 16-in. beams with Poisson's ratio of 1:6

m = constant = 4.73, and
T = 1.23 for d = 3 in., and 1.40 for d = 4 in. (16).

Because of the spread in the results of freezing-and-thawing tests as indicated in Fig. 4, at least ten specimens per variable under test should be subjected to the freezing-and-thawing program and at least five specimens should be used for normal controls. The specimens for each type of test should be so chosen that they will truly represent each batch of concrete used.

Where the objective of the tests is to secure a measure of the resistance of aggregates to freezing and thawing, the following procedure involving freezing in air and thawing in water has proven satisfactory. The rectangular beam specimens should be inserted with the narrow faces as molded uppermost on nonmetallic racks in the cold room. If possible, these racks should be part of the cooling coils so that the temperature gradient in all of the specimens placed in the freezer can be maintained approximately constant. Care should be taken to rotate the position of top and bottom, also the location of the beams after each cycle of freezing and thawing. A suitable fan for stirring the air in the refrigerator room is an additional aid in securing uniform temperature control. The rate of reduction of the temperature in the beams should be determined by measurements with thermocouples inserted at the centers of three or more

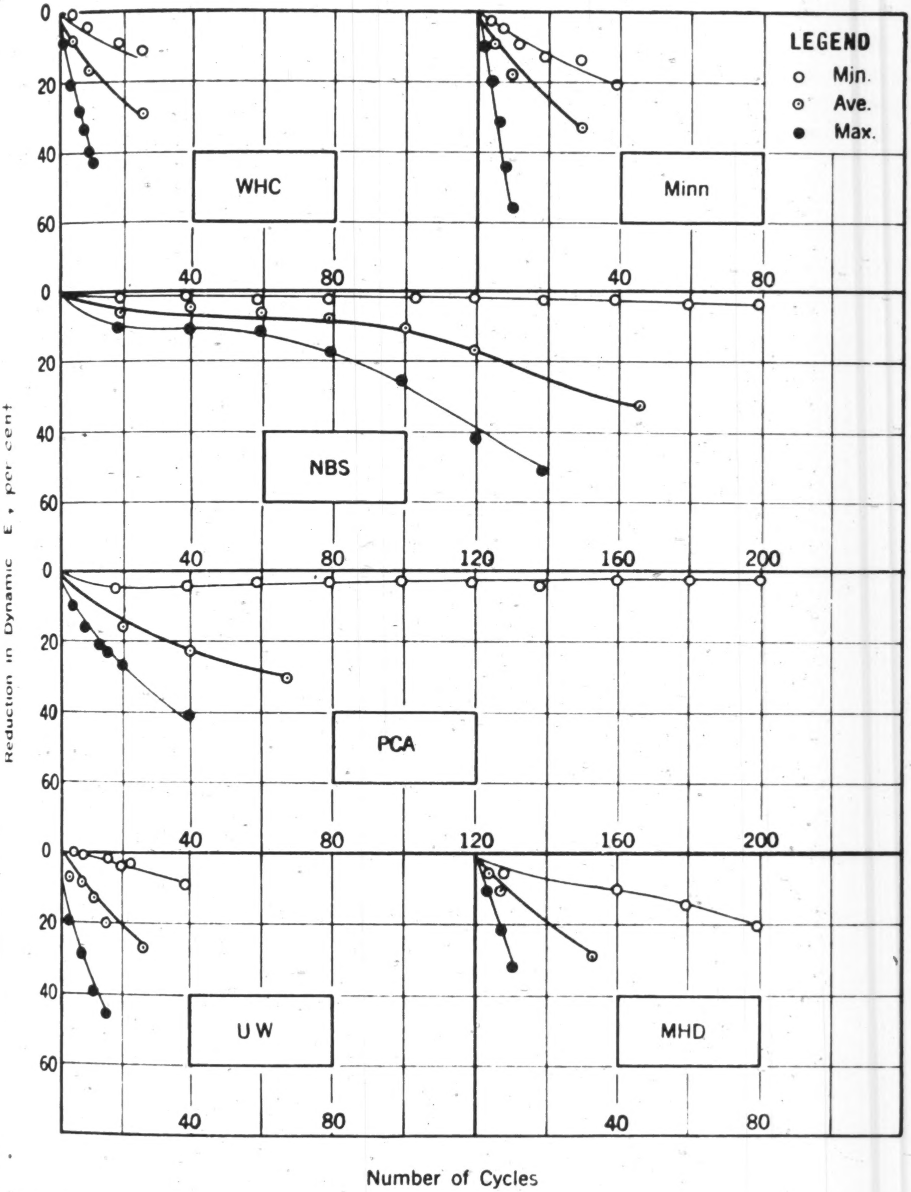


FIG. 4.—Range in Reduction in Dynamic Modulus of Elasticity for Different Numbers of Cycles of Freezing-and-Thawing Tests.

The specimens represented were made at the same laboratory and are supposed to be identical in all respects. The data show the variations obtained when they were tested in six different laboratories. Data taken from report of Committee on Durability of Concrete of the Highway Research Board, 1944.

dummy beams of the same size as the specimens under test. The temperatures of the specimens should be reduced from 40 to 0 F. in not less than 5 and not more than 7 hr., using a minimum freezer temperature of −10 F. If one cycle per day is run, the temperature of the specimens should closely approximate the minimum freezer temperature after the specimens have been in the freezer 16 to 18 hr. It is highly desirable to have temperature-time recording devices for determining the temperature in the dummy specimens and also the freezer temperature during a sufficient number of representative runs to provide information on the uniformity of the temperature controls during the tests.

After the specimens have been in the freezer for 18 hr. they should be removed and immersed for 6 hr. in water maintained at 40 ± 2 F. For concrete of low durability, readings to determine E_d and the specimen weight should be taken on the surface-dried specimens after every cycle. If, however, the durability is relatively high, the interval between readings can be made 5 or even 10 cycles. Inasmuch as the time required for making weight and dynamic modulus readings is small, it is better to take too many readings than too few.

When control beams for strength tests or prisms used for measuring the expansion of the concrete are cured normally, weight and dynamic modulus tests should be run on these specimens from time to time until the end point of the test is reached. If rectangular beams 3 by 4 by 16 in. in size are used in the program, the strength controls should be tested by applying a center load at the middle of the broad face over a 14-in. span. As soon as the E_d of each beam in the freezing-and-thawing tests has been reduced a predetermined amount, say 30 per cent, it should be broken in similar manner. The rate of motion of the movable head of the testing machine in these tests should lie between 0.01 to 0.02 in. per minute.

INDICES OF DAMAGE

In general the rate of deterioration of concrete beams subjected to freezing and thawing is clearly indicated by the reduction in the square of the resonance frequency (f^2) of the beams as the number of cycles of freezing and thawing is increased (11). This follows from the fact that the reduction in f^2 is proportional to the reduction in E_d and, as Reagel (12) has shown, the reduction in flexural strength is a function of the reduction in E_d. Reagel's formula for predicting decreases in moduli of rupture is $R = 6E^{0.6}$, where R and E are percentage reductions in modulus of rupture and modulus of elasticity, respectively. Curves between the reduction in E_d and the number of cycles of freezing and thawing exhibit a very flat slope for specimens of high durability and a very steep slope for those of low durability. It is possible from the average slope of such curves to estimate the relative durability of the concrete under test before the test has been carried to the point where the concrete has begun to disintegrate. Often such information can be secured in 10 cycles and the test then discontinued. *However, until the characteristics of the testing program are well understood, it is advisable to run the tests until the specimens give definite evidence of failure.*

It should be noted, however, that other factors than the rate of deterioration due to freezing and thawing, such as changes in moisture content and temperature changes, have an appreciable effect on E_d of concrete specimens. Also cracks (13) produced in stripping the molds or resulting from mishandling will reduce E_d. On the other hand, since E_d is determined at very low stresses, autogenous healing (**9**) has a more marked effect in restoring E_d than it does on the flexural strength.

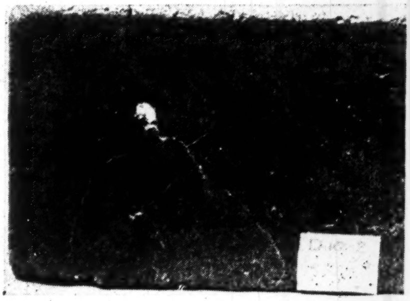
The flexural strength test is a sensitive index (**3**) of the deterioration of concrete when subjected to freezing and thawing. The deterioration of the flexural strength of a concrete with a water-cement ratio of 0.5 by weight is likely to be five times as great as its deterioration in compressive strength. If desired, portions of the beams used for the flexure test may be subsequently tested as modified cubes (**9**) and the compressive strength after freezing and thawing can thus be obtained.

Considerable use has also been made of the loss in weight as an index of deterioration (**14**). It should be borne in mind, however, that this test is of value only when measuring the deterioration of porous, low-strength concretes. Most high-strength concretes lose little weight until they are in the stage of incipient disintegration, at which stage they have lost nearly all of their strength. Furthermore, the loss in weight is much greater when the freezing specimen is in contact with water than when it is in contact with air.

Considerable use has also been made of expansion measurements (**8**) as a means of indicating the deterioration of concrete under freezing and thawing. Although such measurements are nondestructive, they do not afford the quantitative and conclusive measures of the rate of deterioration that result from measurements of E_d.

Concrete specimens of low durability, after a few cycles of freezing and thawing, generally show crumbling at the corners or spalling along the edges. Subsequently transverse cracks appear. The presence of unsound coarse aggregate eventually causes the adjacent surface of the concrete to crack and spall off as shown in Fig. 5. Visual evidences of deterioration are more pronounced when the specimens are frozen in contact with water than when frozen in air.

DETERMINATION OF THE END POINT IN FREEZING-AND-THAWING TESTS



(*a*) After 382 cycles of test of type 1

(*b*) After 380 cycles of test of type 3

FIG. 5.—The Effect of Unsound Aggregate Particles on Frozen and Thawed Concrete Specimens.

Data from University of Wisconsin Tests.

Where the freezing and thawing program is conducted as indicated, that is, the specimens are frozen in air and thawed in water at 40 F., the number of cycles required to reduce the dynamic modulus of elasticity 30 per cent and the corresponding modulus of rupture approximately 50 per cent will vary from five to several hundred (**9**) depending upon the durability of the concrete involved. Hence, for a durable concrete the time required for such tests is excessive. Estimates of the rate of deterioration of a group of durable concrete specimens can be made from either the average slope of curves drawn between the reduction in E_d for a fixed number (10 or 20) cycles or from the actual reduction in E_d after a fixed number of cycles. Then the actual damage can be estimated by determining the average ratio of the flexural strength after the given number of cycles of freezing and thawing to the strength of the normal specimens tested at the time the freezing-and-thawing tests were begun.

SIGNIFICANCE OF FREEZING-AND-THAWING TEST DATA

The accuracy with which the results of an accelerated freezing-and-thawing test predict the behavior of similar material subjected to outdoor exposure is the real measure of the value of such accelerated tests. Unfortunately only fragmentary data are available.

A series of 6 by 18-in. cylinders of 1:3:6 and 1:2:4 (by volume) concrete made at the University of Wisconsin (6) in 1910 were subjected to external exposure with ends resting on the ground. These specimens have been periodically tested in groups of five over a 30-yr. period (15) and the groups have shown a progressive increase in compressive strength including a marked rise in strength between 20 and 30 yr. After 20 yr. of such exposure three specimens of each mix were tested under a very severe procedure of type 1 at the Portland Cement Association Laboratory. No strength tests were made after these accelerated tests but, judging from the loss-in-weight data, 10 to 20 cycles of that accelerated freezing-and-thawing test were far more destructive than the succeeding 10 yr. of exposure on companion specimens. In the latter exposure, our time-temperature records indicate the specimens suffered about 250 cycles of freezing. Other data from our tests on masonry materials lead to the conclusion that the accelerated tests are many times more severe than the action of nature. Tentatively 25 cycles of such accelerated tests may be considered as severe as 10 to 20 yr. of exposure to freezing and thawing in contact with the ground.

NEEDED RESEARCH

1. Further research is required to eliminate the spread between the results of individual specimens supposedly of the same concrete tested in the same laboratory under the proposed procedure of type 3 before that procedure should be proclaimed as standard. Some of the factors which appear to promote such variations in the data for individual specimens are: variations in the degree of saturation of the coarse aggregate at the time of mixing, small variations in the air content of individual specimens due to discrepancies in mixing the concrete and its bleeding after molding, variations arising from variable conditions during the initial curing period, and variations in the degree of saturation of specimens when frozen.

2. Basic research is needed as stated by T. C. Powers (2) to determine the relative influence of the factors causing the breakdown of concrete under freezing and thawing. Successful experimentation to improve the ductility of this brittle material would undoubtedly increase its resistance to cracking, promote greater impermeability and resistance to disintegration under weathering.

3. Experimentation should also be directed towards the perfecting of standard freezing-and-thawing tests suitable for determining the durability of cement pastes, masonry mortars, and concrete products.

4. In order to evaluate properly the results of freezing-and-thawing tests, more information should be obtained from temperature-time measurements in exposed structures. Still more valuable

would be comprehensive information on the behavior of concrete in the several accelerated freezing-and-thawing tests correlated with the behavior of identical concrete subjected to a variety of conditions of severe exposure to the weather.

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DISCUSSION

PREPARED BY CHARLES W. ALLEN[1]

For many years Ohio's experience with freezing-and-thawing tests was limited principally to the testing of concrete cores drilled from pavement and base slabs. The cores were frozen immersed in corrugated copper containers at approximately −20 F. and thawed in water at approximately 70 F. Because of lack of necessary equipment, the specimens were frozen at a local cold storage house. The results were not wholly satisfactory because of the fact that the only means at hand for rating a specimen in the test was by measuring its loss of weight by sloughing as a result of the freezing action. We realized that this means of measurement was very crude, and in some cases wholly unreliable, since a core could fail by cracking and at the same time still lose very little of its weight by sloughing. However, since it was not thought possible to apply the sonic method of measuring the elastic modulus nor, without a great deal of trouble, the length-change method, we believed this to be the only feasible method of measuring the performance of a core specimen in the test.

A very exhaustive cross-file was maintained on test results, every possible classification being made in order to discover the elements responsible for poor performance in the test. Results were classified by general location, specific location, by type of fine aggregate, producer of fine aggregate, type of coarse aggregate, producer of coarse aggregate, and by brand of cements. Separate records were, of course, kept for each class of concrete involved.

Extremely wide variations in test results occurred, some of which were not explainable with the information at hand. In some cases cores failed very early in the test, while the concrete in service performed well. In almost every case, however, when the core withstood long exposure to the test with little effect, the concrete in service performed equally well.

In spite of the apparent shortcomings of the test procedure, the test results on nearly 600 cores at the end of approximately five years began to shape themselves into definite trends. These, when viewed together with service records which were generally corroborative, furnished a promising foundation for further investigation.

In 1945, the laboratory purchased freezing equipment which is now being used to freeze both cores and beams. With this equipment, saturated beams frozen at −10 F. fall from 40 to 0 F. in somewhat less than five hours. Immersed core specimens frozen at −50 F. fall from 40 to 0 F. in approximately seven hours.

One project now under way compares aggregates with good service records with aggregates with questionable service records. For each combination of materials, six 3 by 4 by 16-in. beams were made, each with a corrosion-resistant steel plug in each end. All specimens were cured in the moist room for 28 days, after which three of the six were submerged in water until saturated, and then placed in test as freezing-and-thawing specimens. The remaining three specimens remained in the moist room as controls. The temperature in the freezing box is approximately −50 F., and the specimens are thawed at a temperature of approximately 70 F. in water. These specimens are permitted to remain in the thawing tank approximately twelve hours, then are placed in the freezing cabinet and frozen in air for an equal period. At the end of each ten cycles, each set of three beams is tested for length change, using the corrosion-resistant steel plugs as gage points. The dynamic modulus of elasticity of each specimen is also determined at this time. The apparatus used by our laboratory for this latter determination differs from that described by Mr. Withey in that the specimen is supported upon wooden knife-edges and vibrated vertically by a rod attached to a dynamic speaker located beneath the specimen. The crystal pickup is located directly above the sonic driver and the resonant frequency is determined by means of a cathode ray oscillograph, the maximum amplitude of the resultant wave determining the fundamental frequency. To date, the tests have not been completed. However, the data now available corroborate our previous observations. Questionable coarse aggregates in this series show poorer results than questionable sands.

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[1] Research Engineer, Ohio Department of Highways, Columbus, Ohio.

Our test procedure varies somewhat from that recommended by Mr. Withey. The aggregate used contained approximately the same percentage of moisture as is generally found in the field. It was not possible to control the temperature and humidity in the laboratory as is recommended by Mr. Withey, but the temperature of the concrete was maintained fairly close to 70 F. While we believe that a 40 F. thawing temperature is probably the most desirable for the purposes of the test, the requisite machinery for maintaining a thawing bath at that temperature was not in the beginning available, and further, we felt that we must be positive that each specimen was completely thawed out before the length-change and dynamic modulus tests were made. Lack of space limited the number of active test specimens for each combination to three, but from the results obtained thus far, it is thought that this number is sufficient, since excellent correlation within sets has been obtained. For our purposes, a specimen is considered to have failed in the test when the dynamic modulus has declined by 50 per cent. In certain cases, however, specimens are maintained in the test after this arbitrary end point has been passed.

We should be inclined to question the advisability of placing the test specimens directly upon racks which are a part of the cooling coils. In our opinion such a procedure might subject a localized portion of the specimen to uncontrolled stress, inasmuch as a considerable internal temperature differential must exist in such a case. In our own freezing cabinet the specimens are placed on end between two freezing panels with a small air space on each side.

Of Mr. Withey's article generally, I believe he has performed a signal service in providing the Society with a workmanlike approach to a long-felt need—an accepted procedure for performing freezing-and-thawing tests.

PREPARED BY CHARLES E. WUERPEL[2, 3]

A fundamental concern of engineers engaged in the use and improvement of concrete has long been the problem of resistance to weathering. Although the ability to resist weathering involves complex interrelationships of many properties of the concrete and its component ingredients to many kinds of deteriorating influence; it is the factors involved in measuring resistance to freezing and thawing by a new automatically operated and highly accelerated procedure with which this brief discussion is concerned.

It is patently impossible to duplicate in the laboratory the precise conditions of freezing and thawing to which a particular portion of a concrete structure will be exposed. It is likewise impossible to test full-sized portions of a structure in a standardized weathering exposure in the laboratory. It was believed, therefore, that the most practical procedure would be to devise an apparatus and a method in which standardized test specimens, made from carefully controlled concrete mixtures containing the materials to be used in as nearly as possible prototypal proportions, would be subjected to a standard, automatically operated, reproducible freezing-and-thawing cycle. Other considerations which were believed important and which controlled the establishment of certain elements of the method described herein are:

Time.—It was believed that in order to serve a fully practical purpose results should be available within 28 days after receipt at the laboratory of materials to be tested in freezing and thawing in concrete. The apparatus was therefore constructed to operate automatically and continuously so as to produce one complete cycle of freezing and thawing in a 2-hr. period and eleven such cycles in a 24-hr. period. With an arbitrarily established curing period of 9 days before freezing and thawing is begun, it is possible with this apparatus and procedure to subject concrete specimens to 200 cycles of the freezing-and-thawing test within the desired 28-day period.

Specimen.—The type of specimen selected for use with this apparatus is a beam $3\frac{1}{2}$ by $4\frac{1}{2}$ by 16 in. This specimen is well adapted to rapid and accurate determinations of dynamic modulus of elasticity by vibro-electronic apparatus, can be made from concrete mixtures containing aggregates up to 1 or $1\frac{1}{4}$ in. in size, and allows a maximum number of specimens to be tested in a given cubic volume of space.

Moisture content.—Recognizing that the resistance of hardened concrete to freezing and thawing decreases with increase in moisture content, it followed that the test could be accelerated and made increasingly reproducible by maintaining a high degree of saturation in each specimen. Accordingly, it was decided to immerse the specimens in water and to keep them immersed throughout the test.

The major apparatus which was constructed in accordance with the foregoing concepts consists essentially of three insulated tanks; one for the freezing fluid, one for the thawing fluid, and one for the specimens; with means for cooling the freezing fluid and heating the thawing fluid; and the necessary pumps, valves, and controls to circulate the solutions through the specimen tank at the required temperatures, time intervals, and rates of flow. The particular apparatus referred to herein will reduce the temperature of 102 specimens to

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[2] Chief, Concrete Research Div., Waterways Experiment Station, Corps of Engineers, War Dept., Clinton, Miss.

[3] Based on a paper by the writer and H. K. Cook entitled "Automatic Accelerated Freezing-and-Thawing Apparatus for Concrete," *Proceedings*, Am. Soc., Testing Mats., Vol. 45, p. 813 (1945).

0 ± 2 F. in 1 hr. and raise their temperatures to 42 ± 2 F. in the next hour. It is electrically operated in all details and will automatically repeat that cycle for any desired period of time without attention or will cease to operate after a desired number of such cycles.

A solution consisting of 60 per cent specially denatured ethyl alcohol and 40 per cent water is used for both the freezing and the thawing fluids. The freezing fluid is kept at a temperature of -20 F. and the thawing fluid at +60 F. A total of 900 gal. of fluid is used. Refrigeration is provided by a 15-hp. condensing unit with a capacity of 69,000 Btu. per hour. The thawing fluid is heated by twelve 1000-w. immersion heaters. Circulation of the fluids is accomplished by two centrifugal pumps driven by 1-hp. motors and controlled by motorized valves. Operation of all valves and pumps is controlled by a seven-circuit timer in sequence with a predetermining counter. The tanks are equipped with liquid-level control probes to prevent overflowing. The temperature of the fluids in all three tanks is registered by a multi-pen recording thermometer and the temperature of the interior of a typical specimen is registered on a recording potentiometer.

The specimens are placed vertically in containers, water is added to cover the specimen, and the specimen containers are placed in a rack. Four racks each holding 24 specimens are used; the remaining six containers are placed in the tank individually. The specimens are normally removed from the apparatus for determination of fundamental frequency after 3 and 10 cycles, and thereafter at intervals of approximately 27 cycles. Fundamental frequencies so determined are calculated as percentages of the frequency at the beginning of the test and expressed as relative dynamic modulus of elasticity. When the relative modulus of any specimen has decreased to 50 per cent, the test is discontinued.

The apparatus described above was completed in February, 1945, and was in continuous operation at the Central Concrete Laboratory until August, 1946, during which time some 3000 specimens were tested.[4] It was then dismantled, transported to, re-erected and placed in use at the Concrete Research Division, Waterways Experiment Station, Clinton, Miss. The apparatus has been found to operate successfully and to give results of a degree of reproducibility which appear to offer means of conducting accelerated durability tests in a period suitable for "acceptance" determinations of concrete materials. Such a method of test is believed to be preferable to other available methods because it permits *materials for concrete* to be tested *in concrete* in a highly accelerated, automatic, and satisfactorily reproducible manner.

An apparatus of considerably greater specimen capacity, but otherwise similar in operation to that described in this paper, is to be constructed in the near future. The new apparatus will be made to accommodate a number of different sizes of specimen as well as a greater number by sectioning the specimen compartment and regulating the flow of the freezing-and-thawing solutions.

The accelerated procedure for freezing and thawing has been adopted for use by the Corps of Engineers in the evaluation of air-entraining admixtures and its use in a current research program gives promise of providing a standardized procedure for the evaluation of the durability of aggregates when tested in hardened concrete.

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[4] A majority of the tests conducted are described in an article by the writer entitled "Laboratory Studies of Concrete Containing Air-Entraining Admixtures," *Journal*, Am. Concrete Inst., February, 1946.

Prepared by W. H. Price[5]

Accelerated freezing-and-thawing tests of concrete have been conducted by the Bureau of Reclamation laboratories in Denver for thirteen years for comparing aggregates, cements, admixtures, and methods of curing. The test procedure employed is governed to a large extent by the factors being studied, but in general, 3 by 6-in. cylinders containing $\frac{3}{4}$-in. maximum aggregate concrete of about 0.53 water-cement ratio are employed.

The specimens to be tested are placed in water, within flexible rubber tubes, and are frozen in cold circulating brine at a rate such that their centers reach 15 F. in 1 hr. and 30 min. from an initial uniform temperature of 70 F. Thawing to 70 F. is accomplished in the same elapsed time by placing the tubes in running tempered tap water.

The apparatus and procedures will be described, followed by a discussion of the significance of the test results.

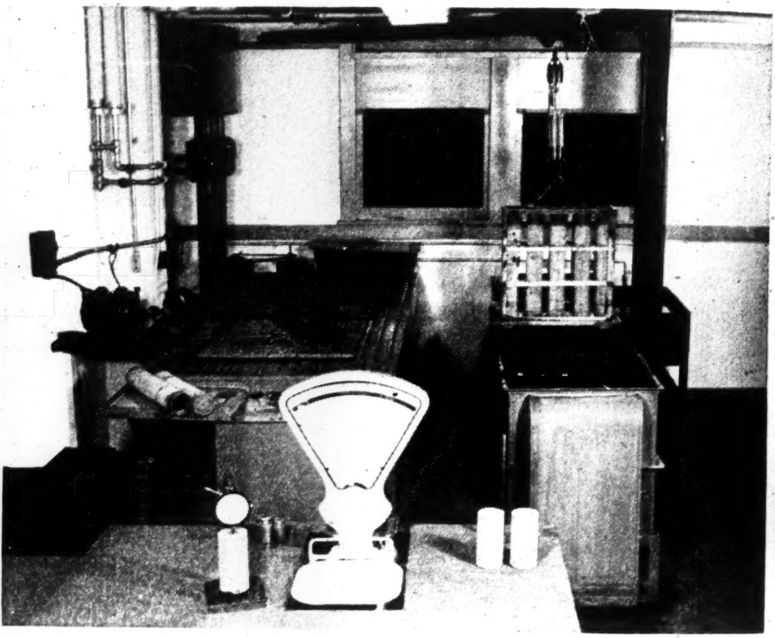

Fig. 6.—Equipment Used in Freezing-and-Thawing Tests on Concrete.

Equipment and Test Procedure

The Freezing Cabinet:

Figure 6 shows the freezing cabinet which consists of a galvanized iron tank, 2 ft. high by 2 ft., 6 in. by 7 ft., 10 in. insulated with 6 in. of cork board and incased in a wooden framework. The tank is divided into five compartments, approximately 1 by 2 ft. in area.

The tank is partially filled with a calcium chloride brine which rises, when the specimens are added, to a depth exceeding the level of the tops of the specimens but lower than the tops of the specimen containers.

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[5] Materials Engineer, U. S. Bureau of Reclamation, Denver, Colo.

The calcium chloride brine is cooled by two sets of rectangular cooling coils connected in series, the coolant, ammonia, entering the top of each coil.

The brine is circulated by means of two turbocirculators submerged in the brine at opposite ends of the cabinet.

An 8-ton ammonia compressor supplies the necessary refrigeration. The temperature of the brine is automatically controlled to 5 ± 5 F. by a system which depends on the pressure of the ammonia in the cooling pipes to activate a mercury switch which regulates the compressor.

A resistance thermometer placed in the brine and connected to a recorder keeps a continuous record of the brine temperature.

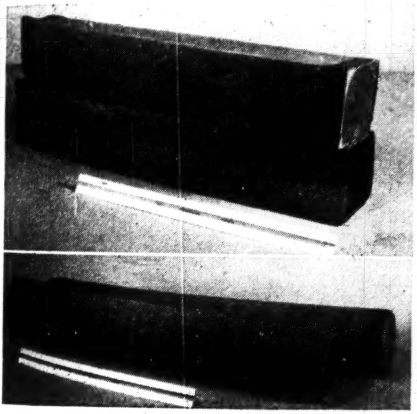

FIG. 7.—Flexible Rubber Containers Used in Freezing-and-Thawing Tests.

The Thawing Tank:

The thawing tank, also shown in Fig. 6, is a galvanized iron tank, 2 ft., 2 in. by 9 ft., 8 in. by 2 ft., 3 in. high, of sufficient capacity to hold 300 specimens. Tap water, tempered to 70 F., enters at two points through the bottom of the tank and is drained at one end of the tank through an overflow outlet. The containers are completely submerged in the thawing water, which flows at a rate sufficient to maintain a uniform temperature.

Rubber Specimen Containers:

The specimen containers shown in Fig. 7 are flexible rubber tubes, $3\frac{1}{8}$-in. inside diameter and $22\frac{1}{2}$ in. in height, with wall thickness of $\frac{1}{8}$ in. The tubes are open at the top, have a $\frac{3}{4}$-in. rubber plug in the bottom, and accommodate three 3 by 6-in. concrete cylinders placed end to end. The rubber used is similar to automobile tire tube stock with a smooth surface inside. A few flexible rubber containers of $3\frac{1}{16}$-in. square cross-section and 22 in. in height are also in use as containers for concrete bars 3 by 3 by $16\frac{1}{4}$ in. in size.

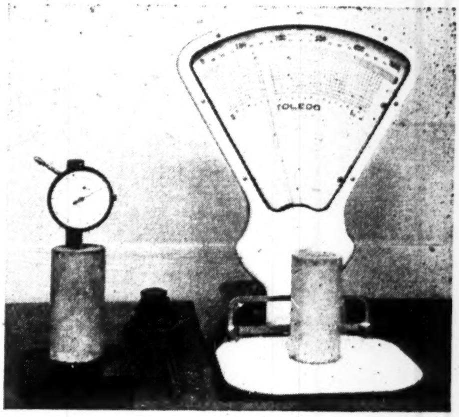

FIG. 8.—Comparator and Scales. Length changes are read to 0.0001 in.

Specimen Racks:

Galvanized iron racks, which will hold ten rubber specimen-containers, are $9\frac{1}{2}$ by 23 by 23 in. in height, and are equipped with wire handles to facilitate handling by an overhead electric hoist. These wire handles are designed to fail under a small load so that jamming of the rack in the brine cabinet during hoist-ing cannot damage the cabinet or hoist. The racks consist merely of iron frameworks which offer little impedance to the circulation of the brine about the containers.

Comparator and Scale:

The comparator (see Fig. 8) used for measuring length changes consists of a steel base with a vertical post to which a movable arm holding a Federal dial reading to 0.0001 in. with a range of 0.4 in. is attached. An anvil in the base receives the gage points or inserts in the specimen. By placing the specimen between this anvil and the plunger of the dial, the length is read directly, for comparison with a standard Invar bar. The dial has a lifting lever to increase the speed specimens can be read.

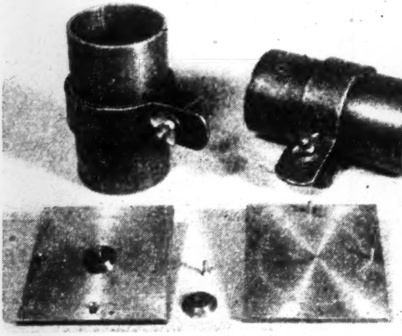

FIG. 9.—Metal Molds for 3 by 6-in. Concrete Cylinders.

A Toledo Scale of 30-lb. capacity and accurate to 1 g. is used to weigh all specimens.

Test Procedure:

The 3 by 6-in. test cylinders are cast with inserts in both ends so that expansion can be determined, Fig. 9. In general the aggregates are in a room-dry state when used and adjustments are made for absorption. Where information is desired in a short period of time, the specimens are cured in the fog room for only 14 days before starting the freezing and thawing. Curing periods of 180 days and longer are employed for studying curing methods and for other research investigations. Fog-cured specimens are soaked in water for 24 hr. and specimens which have been allowed to dry are soaked for at least 96 hr. before starting the cycles of freezing and thawing.

The racks holding the rubber-incased specimens are interchanged between the freezing tank and the thawing tank by means of a hoist and only ten cycles of freezing and thawing are accomplished in a five day week. The fifth day is taken up in weighing and measuring the specimens for weight changes and expansion. Before length measurements and weight losses are determined, the specimens are brushed off and then are allowed to drain for about 5 min. The specimens are replaced in the rubber containers in an interchanged position and remain in the thawing tank over Saturday and Sunday.

An automatic freezing-and-thawing apparatus is now being constructed in which the specimens will be frozen and thawed without being moved and which will permit 12 cycles in 24 hr. Whether it is used at this fast rate of freezing and thawing will depend on the results obtained.

DISCUSSION

Expansion due to freezing and thawing is used as an early indication of failure. A loss of 25 per cent by weight is used as a measure of final failure (see Fig. 10). Visual examination and loss in elasticity are also employed in the evaluation of the test results, but it has been found that with some concretes there is a gain in elasticity for a considerable period after the freezing and thawing has progressed.

Because of the additional information which can be obtained through sonic modulus determination, however, consideration is being given to the use of a bar-shaped specimen 3 by 3 by 11½ in. in size to replace the 3 by 6-in. cylinders now employed.

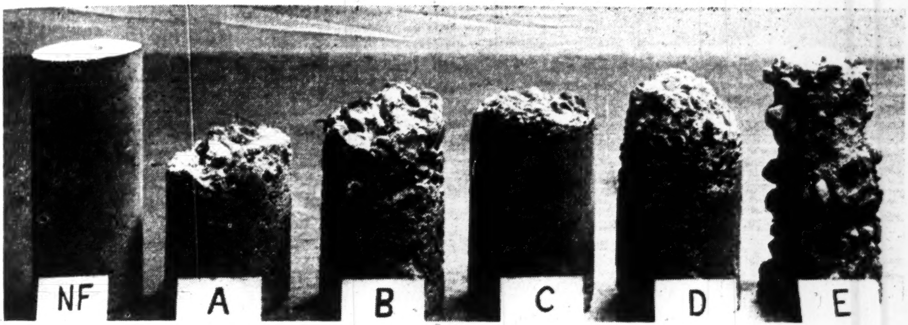

FIG. 10.—Typical Types of Failure (25 per cent weight loss).

Because of the many factors which influence the resistance of concrete to freezing and thawing such as the type, brand, age, and fineness of cement, the degree of saturation of the aggregates and concrete, the length and method of curing, the mix proportions, the temperatures employed in freezing and thawing, etc., a wide variation in results might be expected unless all operations and materials involved in the tests are rigidly controlled. A coefficient of variation of 9.8 per cent was obtained in the cycles of freezing and thawing required for a 25 per cent weight loss for 30 specimens made from ten batches of concrete during a three-week period, using the same materials and proportions and tested under the same conditions. This variation is not considered good when compared to a value of 4.6 per cent for the compressive strengths obtained on companion specimens. A within-batch coefficient of variation of only 1 per cent was obtained in the freezing-and-thawing tests due to the fact that measurements are made at 10-cycle intervals.

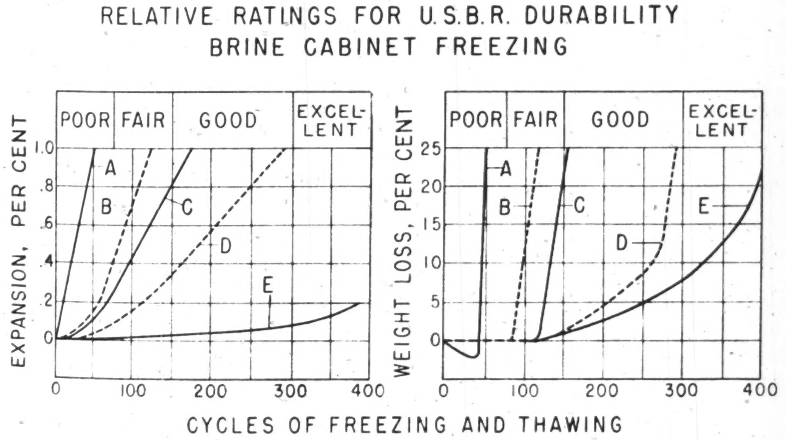

FIG. 11.—Typical Expansion and Weight-Loss Curves for Concrete Durability Specimens.

Criteria for correlating the results of the tests with service performance have not been definitely established. There have been cases where concrete made with aggregates judged suitable by all standard tests, such as sound quartz, failed in comparatively few cycles of freezing and thawing. Others, in which the aggregate was judged poor, stood up remarkably well in this test. These results may only indicate that our methods of judging aggregates should be revised. Surely we should have different standards for mild climates as compared to severe climates.

From information gathered on concretes which are standing up well in service and those which are failing because of weathering, it has been concluded that a concrete which withstands 150 cycles of freezing and thawing in water before losing 25 per cent of its weight is sufficiently durable for severe climates.

The curves shown in Fig. 11 are typical expansion and weight-loss curves for concrete durability tests. The concretes used in these particular tests contained different cements but were all made with aggregates from the same source with the parts of aggregate to cement held constant. The concrete represented by the first curve is rated as being poor. There is some question as to the suitability of the concrete represented by the next two curves. These two concretes would require richer mixes for suitable performance. The last curve is for an air-entrained concrete, containing about 2 per cent air. Note low expansion after 400 cycles. These concretes were all cured for 90 days in a fog room at 70 F. before starting the freezing-and-thawing tests. These results are interesting because if this aggregate were used with the cement represented by the curve on the left it would have been rejected, whereas it would have been accepted if it had been tested with the cement represented by the cement on the right.

COLD ROOM

In addition to the tests already described, 3 by 6-in. cylinders and odd shaped specimens are tested in a cold room.

The cold room is 6 ft., 10 in. by 7 ft., 4 in. by 6 ft., 5 in. high. The room is constructed of wood and is insulated by a 4-in. thick studded partition filled with ground cork and an outer wall of hollow tile blocks 6 in. thick. A temperature of -10 F. is maintained by circulating the air in the room by means of a centrifugal fan through a bank of cooling coils carrying ammonia. The fan is driven, via a shaft through the wall, by an electric motor outside the room. A permanent record of room temperature is kept on a recorder which is operated by a resistance thermometer in the cold room.

The ammonia coils are defrosted every 24 hr. by stopping the flow of ammonia through the coils, then spraying with cold water from a perforated pan. The water and melted frost is caught and drained from a sloping pan under the coils. While defrosting, the blower fan is stopped and the door of the room is kept closed. In this way defrosting can be accomplished without raising the air temperature in the room more than 10 F.

The cold room contains several tables to facilitate the handling of specimens and apparatus in the room.

Procedure:

Specimens to be frozen in air are placed on the truck near the end of the working day, the truck is pulled into the cold room, and allowed to remain overnight. The following morning the truck containing the frozen samples is pulled from the cold room and the specimens are placed, manually, in water in the thawing tanks. At the end of the working day, the samples are replaced on the truck and returned to the cold room for the next cycle. A cycle consists of freezing in air at −10 F. for 18 hr. and thawing in water at approximately 50 F. for 6 hr.

In some instances, specimens frozen in the cold room are subjected to freezing in water-filled metal pans at an air temperature of −10 F. for 18 hr. and to thawing during the day for 6 hr. by placing the specimens into circulating water.

After every ten or twenty cycles, depending on the durability of the specimens, the lengths and weights of the specimens are determined. On specimens which do not contain inserts, such as cores from a concrete structure, durability is determined on a weight basis only.

Freezing in air is much less severe than in water even though the specimen is saturated when frozen, and freezing in water exposed to cold air is less severe than freezing in water in contact with brine. It is probable that the slower freezing cycle of the cold room has some bearing on this apparently greater resistance.

PREPARED BY RAYMOND E. DAVIS[6]

Investigations to determine the effect of freezing and thawing on weathering resistance and other properties of concrete were begun at the University of California in 1932. Various schemes for rating the effect of freezing and thawing have been employed, including visual inspection, change in compressive strength, decrease in modulus of elasticity as determined by static loading, change in modulus of elasticity by the sonic method, loss in weight, and increase in length.

Specimens for freezing-and-thawing tests have included 2 by 4-in. mortar cylinders, 3 by 6-in. concrete cylinders, 6 by 12-in. concrete cylinders, and 2 by 2 by 10-in. concrete bars.

EARLY INVESTIGATIONS

In some of the earlier tests, the specimens were subjected not only to freezing and thawing but also to alternations of wetting and drying. Variables in the cyclic treatment have included minimum temperatures varying from −18 to +23 F. and maximum temperatures varying from 70 to 160 F. Specimens have been frozen both when surrounded with air and when surrounded with a thin layer of water, and specimens have been saturated both with water and with a solution of calcium chloride.

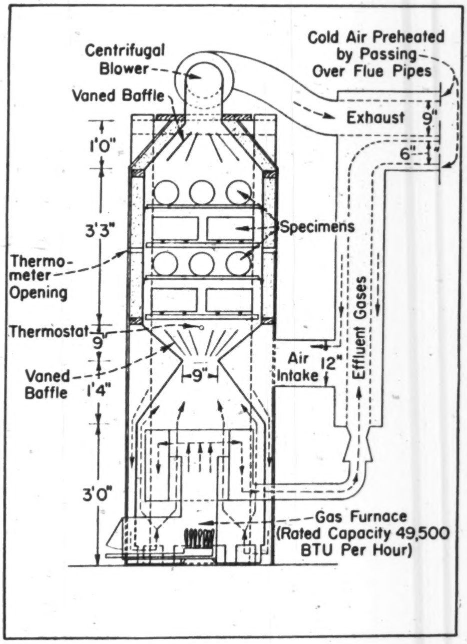

FIG. 12.—Hot-Air Drier.

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[6] Director, Engineering Materials Laboratory, University of California, Berkeley, Calif.

For the early investigations made on 2 by 4-in. mortar cylinders and 6 by 12-in. concrete cylinders, the usual practice was to start the cyclic treatment at the age of 28 days. Half the specimens of each group were continuously stored under standard curing conditions, while the remainder were being subjected to freezing-thawing-wetting-drying cycles. The special oven in which the drying operation was performed is shown in Fig. 12. At the age of 3 months, all specimens were tested to determine their static modulus of elasticity and compressive strength.

For 2 by 4-in. mortar cylinders, the treatment consisted of sixty 24-hr. cycles, as follows:

1. Soaking in water at 70 F. for 10 hr.
2. Freezing in air of a refrigerator room at 23 F. for 4 hr.
3. Soaking in water at 70 F. for 2 hr.
4. Drying in an air blast at 160 F. for 8 hr.

For 6 by 12-in. concrete cylinders, the treatment consisted of thirty 48-hr. cycles, as follows:

1. Soaking in water at 70 F. for $14\frac{1}{2}$ hr.
2. Freezing in air at 23 F. for 10 hr.
3. Soaking in water at 70 F. for $7\frac{1}{2}$ hr.
4. Drying in an air blast at 160 F. for 16 hr.

The specimens were rated by comparing the compressive strength and static modulus of elasticity of the specimens subjected to the cyclic treatment with the strength and modulus of corresponding specimens continuously moist-cured at 70 F.

It was found that excellent correlation existed between reduction in compressive strength and reduction in modulus of elasticity, though on a percentage basis the reduction in modulus produced by the treatment was generally considerably greater than the reduction in compressive strength. While in nearly all cases a reduction in the modulus of elasticity occurred, in several instances, particularly for low-heat cements, the compressive strength of the treated specimens was greater than that of the untreated specimens.

LATER INVESTIGATIONS

Beginning in 1935, freezing-and-thawing tests were made on 3 by 6-in. concrete cylinders which had been standard-cured up to the age of 5 months. These specimens were subjected to thirty 24-hr. cycles of freezing and thawing, each cycle divided into freezing in water, at 0 F. for 16 hr. and soaking in water, at 70 F., for 8 hr. The containers for the specimens were individual tin cans, $3\frac{1}{4}$ in. in diameter by $6\frac{1}{8}$ in. long, filled with water. After each cycle, the specimen was inverted. The specimens were wire-brushed after each 10 cycles. At the end of the 30 cycles of treatment, each specimen was rated as to appearance, by three independent observers, and its loss in weight and compressive strength were determined. The strength was compared with that of corresponding specimens which had been continuously moist-cured under standard conditions. The number of cycles was not sufficient to produce appreciable losses in weight, and there was little difference in condition as rated by visual inspection. All specimens were of a relatively rich mix (1 : 5.6 by weight), the variable being the type of cement. The ratio of the compressive strength of treated specimens to that of corresponding untreated specimens varied from 0.65 to 1.00.

In 1939, the practice of quick freezing at −18 F. was begun. The concrete specimens were 3 by 6-in. cylinders containing end gage plugs for measuring

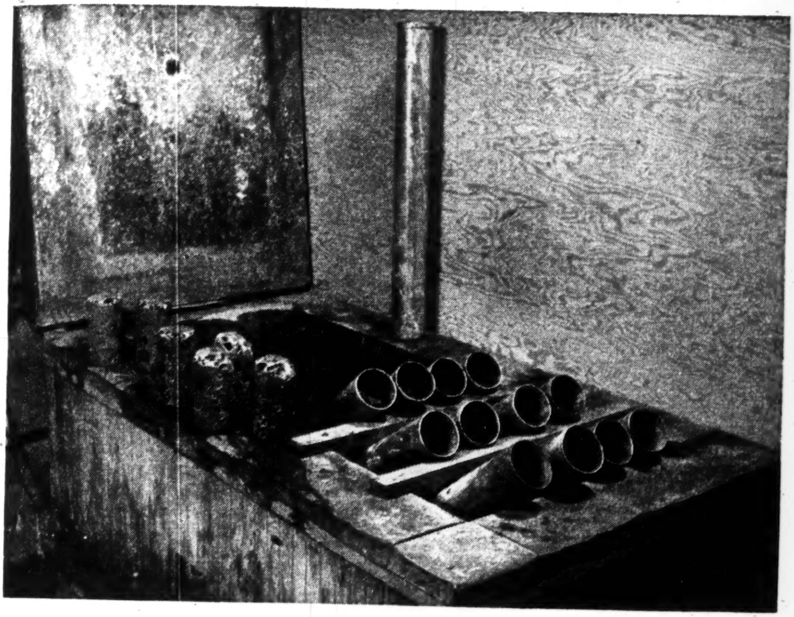

FIG. 13.—Thawing Tank.

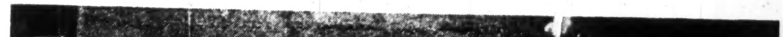

FIG. 14.—Freezing Unit.

changes in length. The treatment consisted of two 4-hr. cycles a day for six days a week, freezing at −18 F. for 2 hr. and thawing (as described later) at 70 F. for 2 hr. At night and on Sundays, the specimens were stored in water at 70 F. Later, the daily treatment consisted of four 2-hr. cycles, freezing at −18 F. for 1 hr. and thawing at 70 F. for 1 hr.

The last-described cyclic treatment is the one generally employed at the present time, tests being started at the age of either 3 or 5 months. After water-soaking, the specimens are placed in a sheet-metal container, $3\frac{1}{16}$ in. in diameter by 30 in. long, three specimens to each container. There is no water surrounding the specimens. The container is then placed in a bath of circulating brine at −18 F. After each period of freezing, the containers are removed from the brine bath, and the specimens are thawed by immersing the containers in water at 70 F.

Figure 13 shows a group of the containers, 12 of which are immersed in the water bath. Figure 14 is a photograph of the brine-tank installation. The capacity of this installation is 150 of the 3 by 6-in. cylinders.

At the end of each 12 cycles, the specimens were weighed, inspected for visual indications of disintegration, and observed for length changes by means of a dial-gage extensometer. The tests were continued until the specimens exhibited a 25 per cent loss in weight. The number of cycles required to produce increases in length of 0.5, 1.0, and 1.5 per cent were observed, as were also the number of cycles to produce losses in weight of 5, 15, and 25 per cent. Also, the number of cycles necessary to produce an appearance rating of 5 was determined, using a rating of "Excellent, 9; Good, 7; Fair, 5; Poor, 3; and Very Poor, 1." Fair correlation was found to exist between the number of cycles required to produce 25 per cent loss in weight and that required to produce 1 per cent increase in length. Also, there was found fair correlation between the appearance rating of 5 and the loss in weight of 15 per cent. The specimens were considered as having failed in freezing and thawing when the loss in weight reached 25 per cent.

In recent investigations, length changes have not in general been observed, and the rating as regards freezing-and-thawing resistance has been based on the number of cycles required to produce 25 per cent loss in weight plus visual inspection by three independent observers.

For concretes containing aggregates which have exhibited what is normally regarded as satisfactory resistance to freezing and thawing in field structures, the number of cycles required to produce 25 per cent loss in weight is 100 or more. Corresponding concretes containing air-entraining agents may require more than 600 cycles.

DYNAMIC MODULUS OF ELASTICITY

In 1941, studies were begun to determine the effect of freezing and thawing on the dynamic modulus of elasticity. For these tests the specimens were 2 by 2 by 10-in. concrete bars containing end-gage plugs for measuring changes in length and a metal retainer in the middle of the top surface of the bar by means of which the sonic hammer was directly connected to the bar (Fig. 15). The freezing-and-thawing treatment was the same as that for the 3 by 6-in. cylinders previously described. After every 15 cycles of freezing-and-thawing treatment, observations were made to determine change in length, loss in weight, and dynamic modulus of elasticity. For one group, the freezing-and-thawing treatment was terminated after 100 cycles, when, in addition to determinations of change in length, loss in weight, and dynamic modulus, the static modulus was determined. Comparisons were made between dynamic modulus and static modulus between dynamic modulus and loss in weight, and between dynamic modulus and change in length. As in previous tests, excellent correlation was observed to exist between loss in weight and change in length; but no such degree of correlation was found between loss in weight and dynamic modulus.

At the beginning of the freezing-and-thawing treatment, the dynamic modulus was found to be about 5 per cent greater than the static modulus; after 200 cycles of freezing and thawing, the dynamic modulus was found to be about double the static modulus.

Figure 16 is a photograph of the sonic apparatus used in determining the dy-

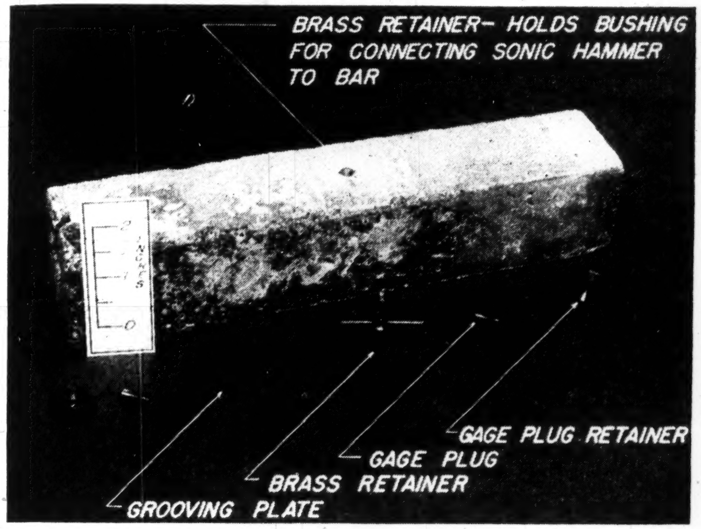
BRASS RETAINER– HOLDS BUSHING FOR CONNECTING SONIC HAMMER TO BAR
2
1
0
INCHES
GAGE PLUG RETAINER
GAGE PLUG
BRASS RETAINER
GROOVING PLATE

FIG. 15.—Dynamic Modulus Specimen.

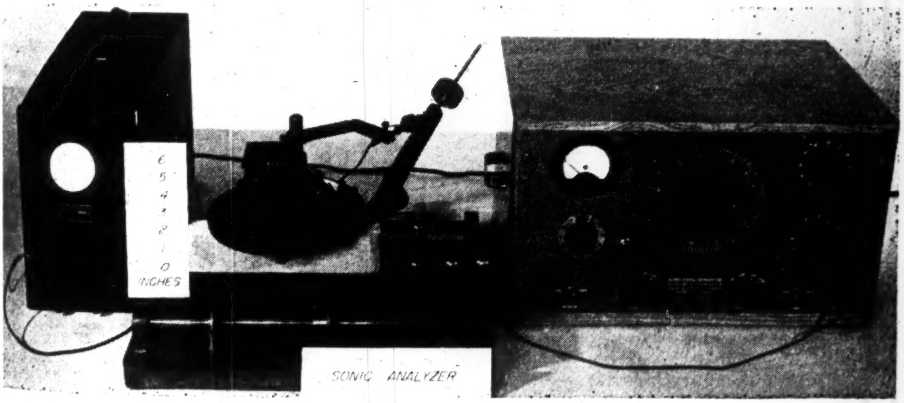
6
5
4
3
2
1
0
INCHES
SONIC ANALYZER

FIG. 16.—Sonic Analyzer.

namic modulus. The bars are supported on wires at the nodal points. The specimen holder is designed to accommodate bars of various sizes and lengths. Vibrations are imparted to the specimen by means of a sonic hammer actuated and controlled by means of an audio oscillator. A phonograph-type pickup is used in connection with an oscilloscope to observe the amplitude of the vibrations. The frequency is controlled by an audio oscillator and is adjusted until the natural frequency of the bar is reached, at which time the amplitude becomes a maximum for the given power input.

DISCUSSION

Reviewing past practices, it appears that either loss in weight or increase in length is a reliable index of the resistance of concrete to freezing and thawing, with little to choose between the two methods. For the majority of concretes tested, substantial increases in length are observed to take place before disintegration becomes appreciable, and for this reason it is believed that where time is limited and a quick estimate of weathering resistance is desired, concrete may best be rated by length changes. There appears to be fair correlation between 0.5 per cent expansion and 25 per cent loss in weight. Generally the number of cycles required to produce 25 per cent loss in weight is roughly double that required to produce 0.5 per cent expansion. For exploratory tests, made for the purpose of selecting materials or combinations of materials which may be expected to give a good account of themselves under severe weathering conditions, it is suggested that in the interests of time and economy, the number of cycles required to produce an increase in length of 0.5 per cent might be made the basis of rating for preliminary selection. Those materials or combinations of materials which were selected on this basis might then be further rated on the basis of the number of cycles required to produce disintegration as measured by 25 per cent loss in weight. The disadvantage of rating by increase in length lies in the larger cost of manufacture of specimens since all such specimens require gage-plug installations.

As a test specimen, the 3 by 6-in. cylinder seems to be satisfactory for most work; and as compared with the 6 by 12-in. cylinder it has the advantage of economy, both in manufacture and in testing.

Because of the time factor involved in making the tests, quick freezing for short periods is desirable, and there appears to be no evidence that the disruptive actions thus produced are not representative of corresponding disruptive actions that may be expected to take place over a much longer period of time under the conditions of the slower freezing rate and the longer freezing period ordinarily encountered in field structures.

If thin metal containers of only slightly larger diameter than that of the specimens are used, there appears to be little advantage in filling the space between container and specimens with water in so far as acceleration of disintegration in concrete is concerned, provided the specimens are immersed in water after each freezing period. The advantage of leaving this thin annular space between the container and specimens filled with air during the freezing periods lies in the quicker thawing of the specimens and elimination of radial compressive stresses which would otherwise be set up within the specimens due to the contraction of the container at temperatures below the freezing point of water and due to the expansion of the ice between the container and the specimens at the lower temperatures. If the space is filled with water and if the walls of the container are thin, which is desirable for quick freezing, the container will progressively increase in diameter due to the expansive forces set up by the ice during each freezing period. In this way a variation of test conditions is introduced which may considerably affect the freezing rate and thus may make questionable the comparability of the results of one series of tests with those of a later series.

Though our investigations have been too limited to justify any final conclusion, the results of tests so far undertaken indicate that, in general, length change is a better criterion to freezing-and-thawing resistance than is change in dynamic modulus.

PREPARED BY A. T. GOLDBECK[7]

In the laboratory of the National Crushed Stone Association, freezing-and-thawing tests on concrete have always been conducted by the use of a cabinet type of freezer. The original machine used was a standard six-compartment ice cream cabinet, and when it became necessary to replace this unit a special cabinet was purchased consisting of a tank 2 ft., 4 in. wide, 3 ft., 4 in. long, and 24 in. deep, surrounded with 6 in. of cork insulation and provided with three separate hinged covers made of metal and filled with 6 in. of cork. The water-cooled compressor is driven by a 1 hp. motor and employs Freon as the refrigerant. This is conveyed through pipes arranged in the form of horizontal coils surrounding the inner vertical walls of the cabinet. The freezing compartment is partially filled with a mixture of water and one of the alcohol anti-freeze liquids commonly used in automobile radiators.

We have used a variety of sizes and shapes of concrete specimens, but in all cases these have been placed in galvanized iron containers, $6\frac{1}{2}$ in. square and 20 in. in height. We have experimented with a variety of freezing-and-thawing conditions, including freezing with the specimens totally immersed and thawing either in the air of the laboratory, in water initially at 70 F. or in water at 40 F.

We have also used the method of freezing the specimens in air and thawing in water at 70 F., and that is our present procedure. The temperature of the alcohol brine, in general, ranges from approximately zero to approximately -10 F. If the load in the cabinet is heavy, the temperature may reach a maximum of $+10$ F.

Our cycle of freezing and thawing consists of approximately 16 hr. of freezing and 8 hr. of thawing. As the result of our experience during the past 15 yr., we believe that the most satisfactory procedure is to place the thoroughly soaked specimens in our immersion cans with no water in the cans except that which drips from the specimens. There is then an air space of about $\frac{1}{4}$ in. surrounding the specimens if they are 6 by 6-in. square beams as sometimes used. The cans are covered with a metal cap having a rubber gasket. This cover has been found necessary to prevent alcohol vapor from condensing on top of the specimens. At the end of the 16-hr. period the cans containing the specimens are removed from the cabinet by the use of a small block and tackle and are placed in water or held under running water until the specimens are free at the bottom and can be slid out into a tank of water at room temperature.

Our freezing specimens are provided with brass plugs set on a 10-in. gage length on opposite sides to permit of taking readings with a Whittemore strain gage. The brass plugs are usually made of $\frac{1}{8}$ or $\frac{3}{16}$-in. brass rod in which grooves are filed for better mechanical bond. These plugs are either molded into the concrete specimens or are set into the concrete at a later stage in holes drilled in the concrete for that purpose. A carbide tipped drill is excellent for this purpose. Very dry neat cement paste is used as the cementing medium.

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[7] Engineering Director, National Crushed Stone Assn., Washington, D. C.

Strain gage readings are taken immediately before the freezing test is started and generally at intervals of ten alternations of freezing and thawing. These readings are made when the specimens are at a temperature of 70 F. as the result of long immersion in water at that temperature. We have found this procedure to be very satisfactory and when the brass plugs are firmly cemented in the concrete we have found that the Whittemore strain gage gives excellent results and the expansion of the concrete produced as the result of freezing and thawing has been found to be an excellent criterion of the effect of freezing on the strength of the concrete. The greater the permanent expansion of the concrete specimen, the greater will be its percentage of loss in beam strength. Our tests are not extensive enough at this stage to tie in the loss in beam strength with the expansion of the concrete, but this relationship could be very readily determined.

As can be seen from this description, the National Crushed Stone Assn. apparatus and method are simple and can be employed in almost any concrete testing laboratory. The method is free of frills and can be performed by a laboratory assistant in a routine way.

In order to increase the capacity of the freezing apparatus, we have lately been using sawed specimens 3 in. square and 12 in. long. Generally, these specimens are sawed from 6 by 6-in. beams resulting from the making of beam tests. Plugs are inserted as usual and strain gage readings are taken to determine the extent of deterioration as revealed by expansion. These specimens are highly satisfactory and have the advantage of quadrupling the capacity of our freezing apparatus. For the sawing of these specimens, we use a standard wood band-saw, except that we have replaced the toothed wood saw with a 1 by $\frac{1}{16}$-in. plain band of soft iron and we have introduced a countershaft for reducing the speed of the saw. The specimen is fed with slight pressure against the band and water is allowed to drip slowly through a stream of carborundum which is carried against and, in part, becomes embedded in the soft iron of the saw. This method is somewhat slow, but nonetheless it is effective. A 6 by 6-in. beam can be sawed in half in from 30 to 45 min., depending upon the hardness of the aggregate.

Referring to Mr. Withey's paper, there is one point I should like to make regarding the mixing of the concrete with the type of mixer illustrated. Such a mixer is difficult to clean thoroughly after the mixture is dumped, and it is surprising how much mortar will remain in the drum. In a recent series of tests it was found that 9 per cent of the total mortar in the mix remained after dumping and partial scraping and consequently the resulting concrete did not have the proportions used in the original batch. When it is considered that it is principally fine material which remains in the mixer, failure to recover it may introduce a large source of error, especially when air-entraining cements are used.

As a result of this experience, I should advocate that the mixer drum be given an initial "buttering" by the use of a small batch of concrete of the same proportions used in making the specimens. Then, when the batch to be used for making specimens is dumped, this batch should be weighed and any deficiency in weight should be made up by scraping the required weight of mortar from the mixer drum. If this procedure is used, the final batch will have more closely the quantities which were originally weighed out than can be obtained by starting the mixing operation with a clean mixer.

SUGGESTIONS FOR INVESTIGATION

One of the disadvantages of the freezing-and-thawing test is the length of time required to obtain significant results. In service, concrete may be subjected to a long period of relatively low temperature with daily rises in temperature which probably thaw a certain depth of the concrete, but this thawing may not extend deep enough to produce complete thawing. What would be the effect on freezing-and-thawing specimens if this incomplete thawing condition were simulated? For illustration, suppose the specimens were first completely frozen for 16 hr., then partially thawed for a relatively short time, say 30 min. or 1 hr. in water at room temperature, then given short cycles of freezing and thawing sufficient only to affect the surface. In this way some three or four or more cycles could be obtained during a day's run. When it became necessary to make sonic modulus tests or expansion measurements, complete thawing would be brought about by longer immersion, after which the short cycles would again be continued.

It has seemed to us that surfaces that are molded against oiled forms have a tendency to be more resistant than trowelled surfaces or surfaces formed by exposing the interior of the concrete by sawing. This point needs further investigation.

In general, it would seem desirable to conduct a freezing-and-thawing test with less labor than is now necessary for the handling of specimens. For this reason, it is believed that there may be considerable merit to the type of machine which permits of freezing and thawing without removal of the specimens from the freezing chamber, except at intervals for the making of the necessary determinations for extent of failure.

PREPARED BY F. V. REAGEL[8]

During recent years several series of tests have been performed in the laboratory of the Missouri State Highway Department in which the resistance of concrete to freezing and thawing has been studied. The objective of these tests was two-fold; first, to develop a test procedure which would give results, accelerated, but comparable to the observed service record of concrete pavements containing different types of coarse aggregates; and, second, to use this test procedure in evaluating various methods of improving the resistance to freezing and thawing of concrete containing certain coarse aggregates.

The test procedure outlined below was adopted several years ago as the procedure showing the best indications of fulfilling the above requirements, and has been used as a standard in all succeeding series. In these series of tests an attempt was made to eliminate the cement and fine aggregate as variables by using cement from one mill and fine aggregate from one source. The fine aggregate was separated on the No. 16 sieve and used in an air-dry condition. The coarse aggregates were separated into three sizes: $1\frac{1}{4}$ in. square opening to 1 in. square opening, 1 in. square to $\frac{1}{2}$ in. square, and $\frac{1}{2}$ in. square opening to No. 10 and used in a saturated condition. The cement factor (1.45), the consistency ($2\frac{1}{2}$ to $3\frac{1}{2}$-in. slump), and the quantity of coarse aggregate (43 per cent) per unit volume of concrete were kept as nearly constant as possible. The beams, $3\frac{1}{2}$ by $4\frac{1}{2}$ by 16 in. in size, have been tested in sets of four. In most series, two sets of beams were made for each variable, and each beam in a set was made on a different mixing date. The concrete was mixed 30 sec. dry and 2 min. wet in an SW Lancaster mixer (one plow used). The beams were stored in the moist room for 7 days, and in water for either 21 or 28 days. When 28 or 35 days old, control beams were broken in flexure, on 14-in. span using center point loading, and beams to be frozen and thawed were frozen in air at 0 F. and thawed in water at 40 F. The beams were placed in the cold room about $\frac{1}{2}$ in. apart on $\frac{3}{8}$-in. dowels which were fastened to wooden racks, the majority of which were supported on cooling coils. No attempt was made to rotate systematically the beams in the cold room, as the rate of cooling has been determined to be approximately the same at all points in the cold room. However, since one $3\frac{1}{2}$-in. face of the beams was nearer the cooling coils, it has been the policy to place the beams on the shelves with the top surface up on odd cycles and down on even cycles.

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[8] Engineer of Materials, Missouri State Highway Department, Jefferson City, Mo.

Originally two equal cycles of 10 hr. in the cold room, and 2 hr. in the thawing tanks were obtained daily. Recently, the time in the cold room has been decreased to 5 hr. for one cycle, and increased to 15 hr. for the other cycle. Work on Sundays and holidays has been eliminated by leaving the beams in the cold room.

A continuous record of the internal temperature of the concrete specimens during the freezing-and-thawing test was obtained by electrically measuring and recording the internal temperature of a control beam.

To trace the progress of disintegration of the beams during the freezing-and-thawing test, the fundamental frequency of vibration of each beam was determined, with electrical apparatus, after specified cycles. Freezing and thawing of an individual beam was stopped when sufficient change in the fundamental frequency had occurred to cause 50 per cent reduction in dynamic modulus or when the beam had been subjected to a specified number of cycles.

The progress of disintegration has also been traced by measuring the change in length of a 10-in. span on both the top and bottom of the specimens. The difference in expansion of the bottom and top of beams has been helpful in studying the effect of different curing procedures and of entrained air.

As a yardstick of relative durability, one coarse aggregate having a good service record has been tested in all series. By plotting percentage loss in dynamic modulus *versus* cycles of freezing and thawing, and comparing other curves with that for the standard coarse aggregate, a fair evaluation of results can usually be made. If there is considerable difference in the flexural strength of the various concretes, then calculated values of the flexural strength *versus* cycles of freezing and thawing are plotted, and these curves are also used in evaluating the results. Also durability factors, which give a numerical value for the curves representing percentage loss in dynamic modulus *versus* cycles of freezing and thawing, have been calculated and used in making comparisons of results obtained. However, none of these methods is considered universally satisfactory.

The results obtained with this freezing-and-thawing procedure have not always been as uniform as desired, nor has the difference in rate of breakdown for the various concretes containing saturated coarse aggregates always been sufficient to justify unqualified conclusions. In general, however, the results obtained with concretes containing normal cements are in fair agreement with the service record of comparable concretes in pavements. This does not necessarily mean that this test procedure can be used to evaluate all suggested methods of improving the resistance of concrete to freezing and thawing. Thus in investigating methods of improving the resistance of concrete to freezing and thawing, it has seemed desirable to use several methods of curing the specimens, as well as to use the coarse aggregates in a saturated and a partially saturated condition. In two recent series in which eleven coarse aggregates were tested with normal and air-entraining cements, four methods of curing were used. These curing methods were:

(*a*) 7 days in the moist room and 28 days in water,

(*b*) 7 days in the moist room and 28 days in an outdoor pit where the tops of the beams were exposed to the weather and the bottoms of the beams were kept wet,

(*c*) 7 days in the moist room and 357 days in water, and

(*d*) 7 days in the moist room and 357 days in the outdoor pit.

Although one of these series has not been completed, certain indications have been obtained from the data collected to date. These are summarized as follows:

1. The method of curing and the degree of initial saturation of the coarse aggregates have a considerable effect, not only upon the rate of breakdown of any specific concrete, but also upon the relative resistance of the various concretes.
2. It may be necessary to use more than one test procedure to obtain sufficient information to determine the relative resistance of several concretes to the freezing and thawing that may occur under service conditions.
3. None of the test procedures used has greatly improved the uniformity of the results obtained with supposedly comparable specimens.
4. Although the methods of measuring the disintegration of the concrete during the freezing-and-thawing test appear to be satisfactory, there is still need for a method of evaluating the results obtained in terms of durability under service conditions.

PREPARED BY BAILEY TREMPER[9]

Freezing-and-thawing tests were started in 1943 in the laboratory of the Washington State Department of Highways, in the thought that such tests should be of assistance in studying an alarming degree of disintegration in the superstructures of a number of bridges of rather recent construction. Various methods that others had used in making this test had been under consideration for several years. An effort had been made to determine what method would correlate best with our field experience. Needless to say, there appeared to be no direct answer to this question. It was thought, however, that freezing in air would be the best approach since this duplicated our field condition. Based on Reagel's excellent paper[10] it was decided to freeze the specimens to 0 F. and to thaw them at a low temperature, around 40 F.

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[9] Materials Engineer, Washington State, Department of Highways, Olympia, Wash.

The large amount of labor involved in handling the specimens during thawing by immersion in water led us to the decision to thaw them under sprays of water. In order to assure equal wetting of all specimens, racks were devised in which the bars were mounted vertically. Figure 17 is a view of such a rack holding thirty 4 by 4 by 20-in. concrete bars. There is a nearly unobstructed space of 1¼ in. between bars. The illustration shows thermocouple leads from one of the central bars which connect with a recording thermometer. Our equipment will handle four of these racks, or a total of 120 specimens weighing about 3500 lb.

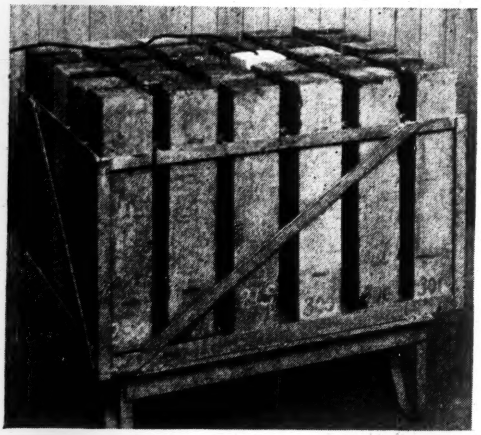

FIG. 17.—Rack for Freezing-and-Thawing Test.

Freezing is accomplished by an overhead forced-draft evaporator. The rapid circulation of air promotes uniform cooling. Thawing is accomplished by moving the racks to an adjoining room, using a hand lift truck. The racks are centered under four shower heads and water is sprayed over the bars in a uniform manner. This procedure therefore constitutes a type of test which differs from the three listed by Mr. Withey.

Figure 18 is a typical 24-hr. record showing two cycles of freezing and thawing. The bars are left in the cold room overnight where they are cooled to a temperature of approximately 0 F. At 8 a.m. they are transferred to the thawing room where, under water sprays, they reach 40 F. in about 1 hr. This is the recorded temperature at the center of the bars. At this time, however, there is a coating of ice about ¼ in. thick over the bars. One hour additional is required to melt the ice. By this time the bars have reached a temperature of about 45 F. Our cooling water comes from an artesian well and has a temperature of about 50 F. It is not recirculated but could be if the cost of water were an item. At 10 a.m. the bars are returned to the cold room. They are lowered to 32 F. in about 1 hr. and reach 0 F. in about 5 hr. At 3 p.m. the second cycle of thawing is started and they are returned to the cold room at 5 p.m. where they remain overnight. Two cycles are completed daily including Sundays and holidays, except that on each seventh day, one cycle is

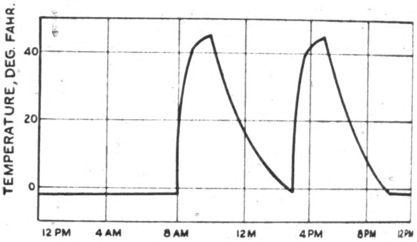

FIG. 18.—Typical Temperature Record.

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[10] F. V. Reagel, "Freezing and Thawing Investigations," *Proceedings*, Highway Research Board, Vol. 20, p. 587 (1940).

omitted while measurements of weight and dynamic modulus of elasticity are being made. Length changes have been measured in smaller specimens of 10-in. gage length, but not in the 20-in. bars we have mostly used.

Soon after we started these tests we made comparisons with the results obtained by the method used by the Missouri Highway Department. The only change we made in our procedure was to thaw the bars by immersing them in water instead of subjecting them to sprays of water. Figure 19 shows the difference in results between the two methods. In our method, the concrete was affected very little by 78 cycles, whereas the Missouri cycle damaged it severely. The difference is thought to lie in the fact that immersion forces more water into the bars because of the slight hydrostatic head to which they are subjected.

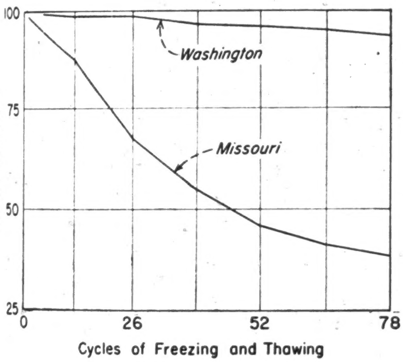

FIG. 19.—Comparison with Missouri Cycle.

Figure 20 shows that, in spite of the apparent mildness of our procedure, we are able to produce severe failure in some concretes. Fifteen cements are represented on this chart, all combined with the same aggregate. Note that the concrete made with five of the cements was decidedly lacking in resistance.

It should be explained that the pretreatment of the specimens represented in this chart consisted of 28 days moist curing at 70 F., 78 cycles of freezing and thawing, then 6 months additional moist storage. None of the specimens was affected significantly in the preliminary run of freezing and thawing, and the slope of the plotted curves indicated that all would remain resistant through a very large number of cycles. We have found this condition frequently in our work. Certain combinations of cement and aggregates that appeared durable initially have failed rapidly when retested after additional moist storage. On the other hand, other combinations have been highly resistant in the second run. Time does not permit going into further details. We have not arrived at a reasonable explanation of the mechanism that takes place.

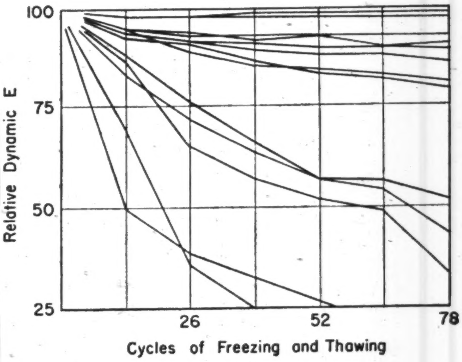

FIG. 20.—Typical Test Results.

In general, the procedure used in our laboratory appears to give better agreement between individual specimens than is reported by Mr. Withey. Probably this is the result of a slower rate of attack in our method. Nevertheless, in about 15 per cent of our tests the spread between individual specimens is of the same order as shown in Mr. Withey's Fig. 4. The reason for the wide divergence among supposedly similar specimens is not understood and it is important that the cause be discovered and eliminated.

In our research work, average values from four specimens from two batches, or three specimens each from a separate batch, have shown concordant trends and have warranted rather definite conclusions. If the test were to be used for specification purposes, however, I agree with Mr. Withey that a larger number of specimens should be used.

Figure 21 shows the degree to which the results can be duplicated. The solid lines represent tests made with three cements, A, B, and C, in 1943. The broken lines represent tests made two years later with cements from the same lots and aggregates from the same pit but sampled at a different time. If the agreement does not seem as good as might be desired, it should be borne in mind that the fine aggregate was not graded exactly the same in both cases, that the water-cement ratio and consistency were not duplicated exactly, and that two years intervened between tests. In each group, the three cements are in the same order and show roughly the same degrees of resistance.

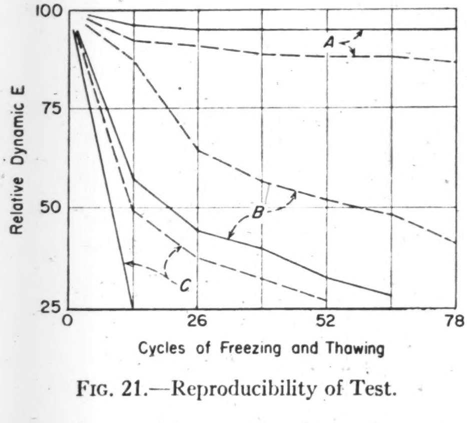

FIG. 21.—Reproducibility of Test.

The question now arises as to how well the cycle duplicates the effects of natural exposure. For the case we have studied, namely, the resistance of small bridge members exposed directly to the weather, it would seem that there is much logic to our procedure. Freezing is accomplished in the medium of air, as are our bridge members. Thawing takes place by sprays of water at relatively low temperature, which is similar to the action of rain or melting snow. The laboratory specimens assume the appearance of the bridge members in many respects. Pop-outs occur when particles of unsound aggregate are near the surface. Patterns of surface cracks similar to, but on a smaller scale than on the larger bridge members, become evident during the tests. Brown deposits develop along cracks in affected laboratory bars and they have much the same appearance as on similar concrete in the field.

Where we have both good and bad examples of bridges containing alkali-reactive aggregates under severe exposure, we have been able to develop similar distinctions in the laboratory. Our tests also show poor resistance for those combinations of cement and nonreactive aggregates that have given poor service in structures. Laboratory bars made with other cements and the same aggregates show good resistance, but unfortunately we do not have equivalent concrete in the field for comparison.

In order better to compare our test results to natural field conditions we have exposed parallel groups of bars to outside weathering in a mild climate and at a location where severe winter conditions prevail. Bars from the same groups have been subjected to the laboratory freezing-and-thawing test. These bars comprise many combinations of cement and aggregates. They have been exposed outside for only two and three winters and there has not been time for much deterioration to take place. The indications to date, however, are such as to inspire confidence in the belief that the laboratory test will predict the future relative condition of those bars that are exposed to severe outside weathering.

In conclusion let me stress that the aggregates used in our tests have not been unsound but on the contrary have been of excellent quality judged by customary standards. The cements used, likewise, have conformed to standard specifications. It has been only with unfavorable combinations of these materials that our freezing-and-thawing test has indicated a lack of resistance that appears to explain our difficulties in the field. To what extent it would aid in solving problems of poor durability due to other causes I am unable to say. I do feel, however, that the method merits trial in other laboratories.

PREPARED BY RUDOLPH C. VALORE, JR.[11]

Freezing-and-thawing studies of concrete have been carried on at the National Bureau of Standards continually for more than 20 yr. These studies fall into general groups: (*a*) attempts to evaluate the effect of the constituent materials or methods of fabrication of concrete specimens on the relative durability; (*b*) studies of test methods and of means of measuring the degree of disintegration; and (*c*) studies of the mechanism of disintegration of concretes under the action of freezing and thawing.

To the construction engineer, "durability" is usually synonymous with the ability of a material in a structure to withstand the destructive effects of weathering. It is usually for the purpose of predicting this ability that freezing-and-thawing tests are made. What is actually determined in the laboratory, however, is the relative ability of specially fabricated samples to withstand the effects of rapid and continuous alternations of artificial freezing and thawing. These effects cannot be expected to be consistently similar to the effects of outdoor exposure. Other weathering processes, such as wetting and drying, and daily cyclical temperature changes without freezing or thawing, also are known to affect concrete. Therefore, the freezing-and-thawing tests purport to measure only one phase of concrete durability. It has not as yet been found possible reliably to estimate durability from an analysis of any of the more easily measured physical properties of concrete.

Freezing-and-thawing procedures presently in use at this Bureau differ widely from those described by Tucker, Walker, and Swenson (**1**)[12] in reporting work done at this Bureau from 1927 to 1931 on the physical properties of cast stone. In that work the specimens were placed in $\frac{1}{4}$ in. of water in trays which were then placed in a room maintained at a temperature of about 10 F. Thawing was performed by immersion of the specimens in water at 70 F. One cycle of freezing and thawing was performed each day and criteria for failure were loss in weight and various types of visible signs of disintegration. The correlation between resistance to freezing and thawing and the several physical properties of the cast stone was described as "very poor."

Subsequent procedures have shown an increasing emphasis upon acceleration of the freezing treatment. Present procedures, described in detail elsewhere in this report, in which specimens are placed in water in rubber containers which are immersed in liquid at temperatures as low as −20 F., bear little resemblance to natural freezing and thawing. This severe treatment is justified for reasons of expedience. The time and the cost would be too great to justify the use of a mild treatment more closely simulating natural processes, especially since the advent of the highly resistant aerated concretes. However, until precise methods are developed for the measurement of those properties of concrete most certainly correlated to durability, test methods must, because of economic expediency, continue to be developed along empirical lines.

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[11] Materials Engineer, National Bureau of Standards, Washington, D. C.

[12] **The boldface numbers in parentheses refer to the references appended to this paper, see p. 1238.**

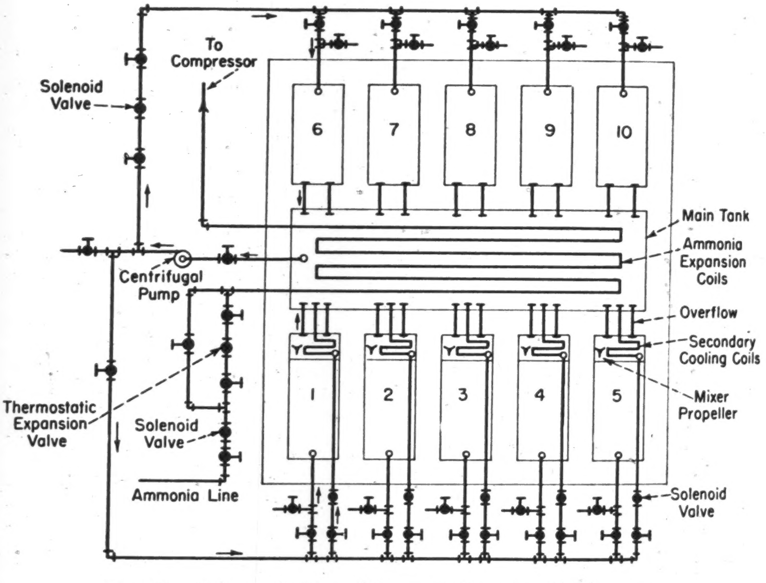

FIG. 22.—Schematic Plan of Concrete Freezing Equipment.

SCOPE OF FREEZING-AND-THAWING STUDIES

Freezing-and-thawing tests at the National Bureau of Standards are made primarily for the purpose of differentiating among the relative durabilities of various cements, aggregates, or admixtures as they appear in concrete, individually or in combination. Tests of concrete containing any material in question are accompanied by tests of specimens containing materials of known performance whenever it is possible to do so.

Studies have been made of test methods, including fabrication and curing procedures, and of the effects of varying rates of temperature change. Fundamental experiments upon the volume changes in concrete during freezing and thawing, as part of a more general study on the mechanics of freezing and thawing, are in progress. In addition, investigations have been made for the purpose of measuring with greater precision the many intended, inherent, and accidental variables in concrete. Among these variables the percentage of saturation, porosity, permeability, quantity of freezable water, air content in plastic concrete, and the specific heat of moist concrete in subfreezing temperatures become increasingly important in freezing-and-thawing work.

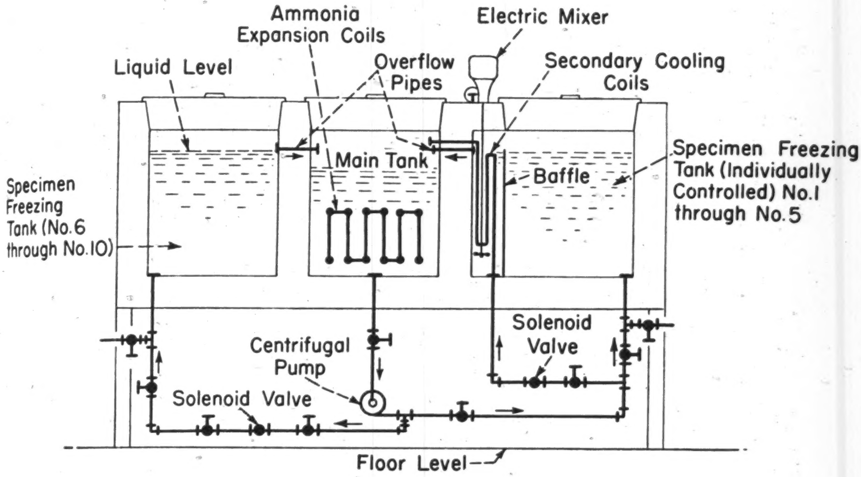
Ammonia
Expansion Coils
Electric Mixer
Liquid Level
Overflow
Pipes
Secondary Cooling
Coils
Main Tank
Baffle
Specimen Freezing
Tank (Individually
Controlled) No.1
through No.5
Specimen
Freezing
Tank (No.6
through No.10)
Solenoid
Valve
Centrifugal
Pump
Solenoid Valve
Floor Level

FIG. 23.—Schematic Elevation, Concrete Freezing Equipment, Showing Main Tank and Two Types of Specimen Freezing Tanks.

FREEZING-AND-THAWING EQUIPMENT AND PROCEDURES

Operation of Refrigeration Equipment:

Equipment in use at this Bureau since 1938 for freezing specimens of concrete is shown schematically in Figs. 22, 23, and 24. Refrigeration is obtained by means of ammonia expansion into coils situated in a main cooling tank. The specimen cooling medium now in use is a nonflammable mixture of ethylene glycol and water, although an alcohol-water mixture was originally and may again be used. The liquid is pumped from the main tank into the piping system, up through the bottoms of the individual specimen freezing tanks, so that it overflows back into the main tank, to complete the liquid circuit. This is shown in Fig. 23. Figure 22 shows the schematic arrangement of the tanks and piping system. Under the arrangement described the temperature of the liquid in the individual tanks varies only with temperature variations in the main tank. This temperature is controlled by means of a thermostatic expansion valve in series with a solenoid valve in the ammonia supply line, as indicated in Fig. 22.

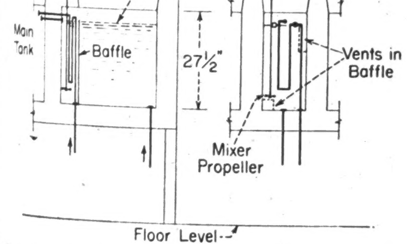
Secondary
Cooling Coils
Overflow
Pipes
12"
Electric
Mixer
30"
12"
Liquid Level
Main
Tank
Baffle
27½"
Vents in
Baffle
Mixer
Propeller
Floor Level

FIG. 24.—Specimen Freezing Tank (Individually Controlled) Schematic Diagram.

An alternative, providing a desirable flexibility of operation, is used in the operation of tanks numbered 1 through 5 (see Fig. 23). Each of these tanks is provided with secondary cooling coils through which the cold liquid, acting as refrigerant, is pumped from the main tank. No liquid is pumped through the individual tank itself, but instead, a definite quantity of liquid is contained in the tank. A vertically mounted electric mixer provides the necessary circulation around the coils and within each tank. The temperature of the liquid in each of these tanks may be automatically controlled by means of a solenoid valve in each secondary cooling coil line operated by individual gas bulb temperature controllers. As ordinarily used, the temperature of the liquid in the main tank is maintained at −20 ± 2 F. and any desired temperature between −10 and +40 F. may be maintained in each of the five individually controlled tanks. This arrangement makes it possible to cool simultaneously several groups of specimens at different rates, and the equipment in general is easily adaptable to experimental work upon variables in freezing procedure.

Thawing is ordinarily done by immersion of the specimens in water in a tank of about 300-gal. capacity, the temperature of which is controlled manually at 70 ± 2 F. by mixing cold water and steam. Circulation within the tank is provided by means of a centrifugal pump. As an alternative, thawing may be done in any of the individual freezing tanks described in the preceding paragraph when thawing temperatures below 40 F. are desired.

Mixing, Fabrication, and Curing of Specimens:

Under present testing methods, moist aggregates are used. The moisture is determined by evaporation, and proper corrections in the mix proportions are made prior to mixing.

Mixing and fabrication of specimens are done in a constant-temperature laboratory maintained at 70 ± 2 F. The materials are mixed for a period of 2 min., allowed to stand for 1 min., and mixed again for 1 min. in a tilted drum mixer of 3 cu. ft. capacity, rotating at 22 rpm. Slump, flow, and density measurements are made upon each batch. Air in the plastic concrete is determined by the Pigman method (2) when it is desirable to measure this property.

The molds, coated with a very thin film of light grease and sealed watertight with a stiff graphite grease, are filled in two equal layers. Each layer is rodded 30 times and each vertical cast surface is spaded; the excess material at the top of the mold is then struck off by using a transverse sawing motion with a $\frac{5}{8}$-in. steel rod. The mold is then covered with damp burlap. The exposed face of the specimen is trowelled smooth after bleeding has ceased. The specimens remain in the constant-temperature room, covered with damp burlap, for 24 hr., after which they are removed from the molds and transferred to a fog room which is also maintained at a temperature of 70 ± 2 F. After 6 days in the fog room, the specimens are removed to a constant temperature, constant humidity room (70 ± 2 F. and

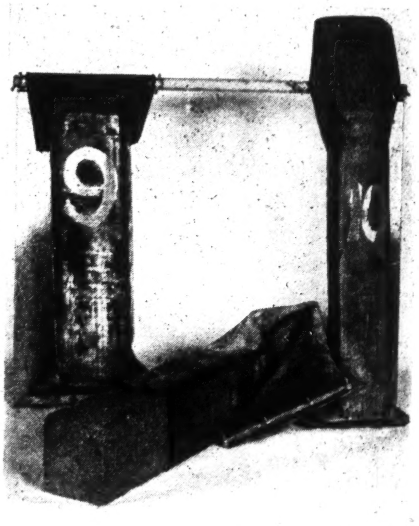

FIG. 25.—Rack for Holding Rubber Bags Containing Concrete Specimens. One bag is clamped shut in the rack as it is during freezing-and-thawing treatments. Another bag stands with its top open. The third bag is shown with specimen protuding. This rack will hold six specimens.

50 ± 5 per cent relative humidity), where they are air-dried for 18 days. They are then placed in water at 70 ± 2 F. for 3 days so that they are ready to undergo freezing-and-thawing treatment at the age of 28 days. This curing procedure, however, may be varied according to the degree of acceleration of treatment desired, as in the case of certain aerated concretes which begin the freezing-and-thawing treatment at the age of 15 days, after having been cured for 13 days in the fog room, followed by immersion in water for 1 day.

Freezing-and-Thawing Treatment:

The type of specimen used in most freezing-and-thawing tests is a 3 by $3\frac{1}{2}$ by 15-in. prism. This prism is encased in a snug-fitting rubber bag of $\frac{1}{16}$-in. thickness (automobile inner tube quality). About one pint of water is added, which is sufficient to cover the entire specimen during test. No water is added, however, if it is desired to test the specimens in a constant-moisture condition. Bags containing the specimens are held vertically in racks in such a way that, when immersed, either in the freezing or the thawing liquid, the external liquid pressure collapses the bag against the contained specimen and forces out the entrapped air. The bags are clamped shut at their tops to prevent the accidental interchange of liquid between the inside and the outside of each bag, as illustrated in Fig. 25. An electric hoist is used to transfer the racks, holding six or eight specimens, between the thawing and individual freezing tanks.

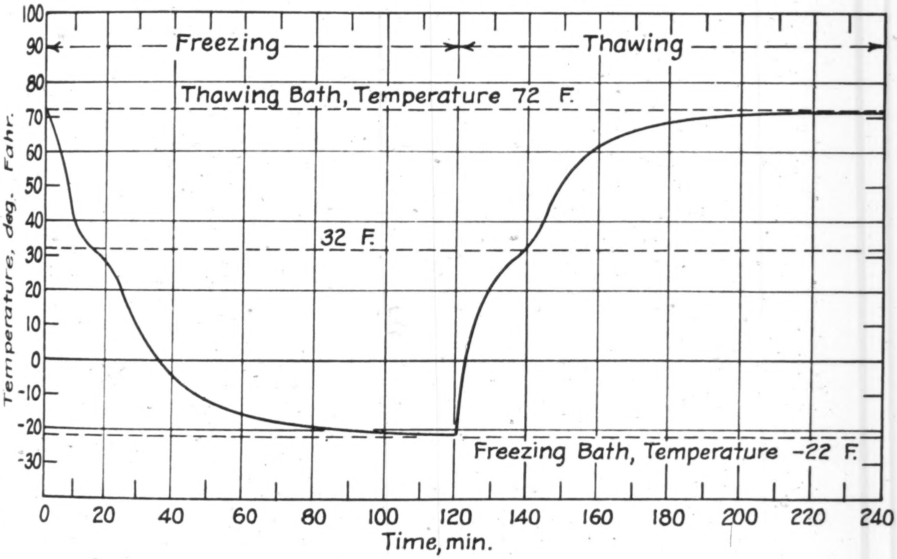

FIG. 26.—Typical Curve for Temperature at the Center of a 3 by $3\frac{1}{2}$ by 15-in. Specimen for On Complete Cycle of Freezing and Thawing.

Other sizes of specimens have been used in freezing-and-thawing work, particularly a $2\frac{1}{16}$ by $3\frac{3}{4}$ by $8\frac{1}{16}$-in. brick-shaped specimen, which is a convenient size for test for air permeability in an apparatus used at this Bureau (3).

The type of freezing-and-thawing cycle used is varied according to the purposes of the tests. A relatively severe treatment (+70 to −20 F. in 2 hr.) is used for comparing the efficacy of air-entraining admixtures or cements in increasing the (laboratory) durability of concrete, since a milder treatment would render prohibitive the amount of time necessary for the accumulation of useful data. A typical curve for the temperature at the center of a 3 by $3\frac{1}{2}$ by 15-in. specimen as it undergoes this severe treatment is presented in Fig. 26. Milder treatments have been used (+70 to 0 F., +70 to +10 F., or +70 to +25 F. in 2 hr.) where aggregates or cements are compared for durability in concrete, since the more severe treatment would break down the materials too quickly to allow precise differentiation to be made.

Normally a specimen undergoes two cycles of freezing and thawing each day by immersion for 2 hr. periods alternately in the cold liquid and in the thawing liquid, and remains over nights and weekends in the thawing liquid.

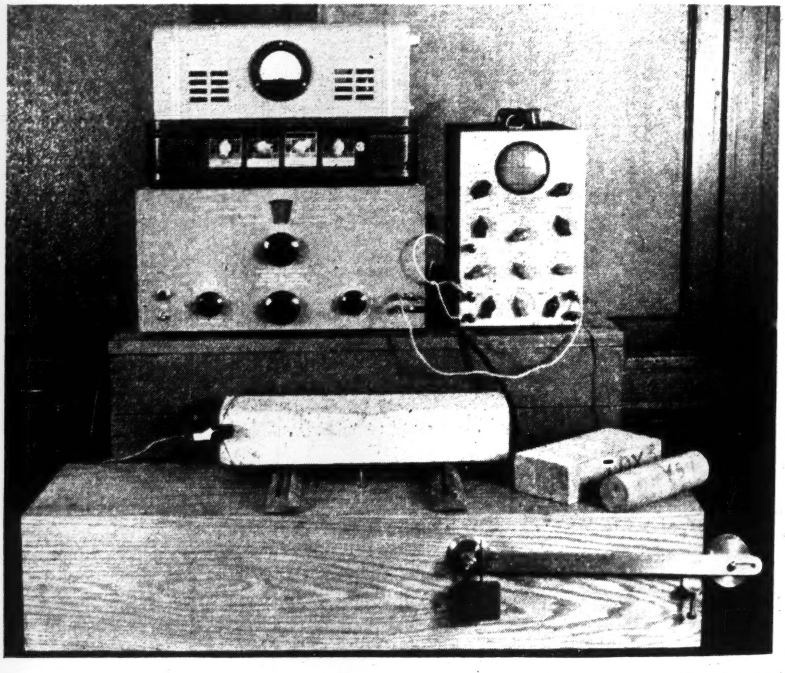

FIG. 27.—Sonic Testing Equipment. Specimen has been moved back so that the driving rod protruding vertically from the box-like mounting that houses speaker-driver may be seen. Adjustment of counter-weight brings driving rod into steady contact with specimen. Variable audio-oscillator, amplifier and controls, a-c. millivoltmeter, and cathode ray oscilloscope are shown. Also shown are other types of specimens used in freezing-and-thawing work.

Determination of the Effects of Freezing and Thawing:

The relative "soundness" of a specimen is determined by obtaining its natural or fundamental frequency of flexural vibration. The specimens are usually vibrated in a direction normal to their trowelled surfaces. Normally, a specimen is removed from test when its dynamic modulus of elasticity, *E*, has been decreased by 30 per cent from the modulus obtained at the start of the freezing-and-thawing treatments, or when it has withstood a predetermined number of cycles (usually 200 or more) without reaching this end point. Dynamic E values are usually not computed, since the percentage change in the square of the fundamental frequency is identical with the percentage change in dynamic E. Examination for visible signs of disintegration is a necessary adjunct to periodic sonic tests, and unusual variations in appearance or weight are recorded into the life history of a specimen.

The sonic testing equipment used, shown in Fig. 27, is of a type commercially available for concrete testing.

PROPOSED CHANGES FROM PRESENT PROCEDURES

In the near future the following changes in procedural detail are planned:

(*a*) The size of the specimen will be changed from 3 by 3½ by 15 in. to the size more widely used in other laboratories, 3 by 4 by 16 in. Molds of the 6-gang type now used will be replaced by single-specimen molds.

(*b*) The specimens will remain in the frozen condition over nights and weekends instead of in the thawed condition. This is a reversion to the practice followed 20 yr. ago at this Bureau, but which was abandoned in more recent years because of the limitations of equipment.

(*c*) The thawing temperature will be changed from +70 ± 2 F. to +40 ± 2 F. and will be controlled automatically rather than manually. It has been shown that, other things being equal, a lowering of the thawing temperature accelerates the decrease in dynamic E (**4, 5**).

(*d*) The number of freezing-and-thawing cycles administered each day will be increased from two to four without changing the freezing bath temperature for the individual type of cycle as used heretofore for any particular type of test. The specimens will remain immersed in freezing or thawing liquids for 1-hr. periods instead of 2-hr. periods. Although in certain severe treatments the centers of the specimens may be at an appreciably higher temperature than the freezing bath after 1 hr. of immersion, the rate of temperature change will not be essentially different from what it is for 2-hr. immersion periods if neither freezing nor thawing bath temperatures are changed. The 1-hr. period is sufficiently long to allow complete thawing of a specimen, even in the more severe types of freezing-and-thawing cycles.

The purpose of increasing the number of cycles per day is to increase the rate of disintegration without increasing the rate of freezing.

In addition to the above-mentioned changes, the following suggestions require further investigation before they may become a part of the freezing-and-thawing procedures:

(*a*) The use of prismatic specimens carefully sawed from large concrete slabs is suggested for certain types of freezing-and-thawing studies where the present type of specimen with its very high proportion of cast (and possibly overworked) surface or "skin" may not be satisfactory. The use of sawed specimens might give greater meaning to absorption and permeability measurements than has been previously derived from them. Such specimens might also provide a sample more truly representative of concrete as it is in a structure than that provided by the presently used type of specimen.

It is further suggested that the effects of freezing a single face, while all other faces of the prismatic specimen are maintained at a temperature above freezing, be studied as an approximation toward natural freezing conditions.

(*b*) Incorporating present procedures into an entirely automatic system would greatly accelerate the testing of concrete for freezing-and-thawing durability, by increasing more than fourfold the number of cycles per unit of time. Automatic equipment, operating on the liquid immersion principle, as described by Wuerpel and Cook (6), and equipment employing a cold air blast freezing treatment and water-spray thawing treatment for uncovered specimens, are being considered. An advantage of automatic methods, apart from savings in labor, would be the employment of lower rates of temperature change, more closely simulating natural freezing, without increasing the time required for completion of tests under more severe treatments now used in which operations are suspended each night and each weekend.

CONCLUSION

There is at present general disagreement among concrete technologists concerning the most satisfactory procedures for making accelerated freezing-and-thawing tests of concrete.

While results usually show broad and general agreement, there is a welter of conflict and seemingly inexplicable divergences in results of tests made upon supposedly similar materials. It is realized that control is necessary in the production and curing of test specimens. But this realization must not lead us to expect to find in concrete a degree of homogeneity and reproducibility that is not inherent in the material. It is felt that our attention should be directed toward evolving methods for the precise evaluation of the variables in concrete that often appear to be so greatly magnified when translated into resistance to freezing and thawing.

One of the aims of research at this Bureau has been the development of rapid and reliable methods for predicting the durabilities of all types of concrete. One method, based upon the volume changes during only one cycle of freezing and thawing, shows promise in early trials. But perhaps it is not inappropriate to hope that we may some day reach the ultimate—in being able to predict freezing and thawing, or even "weathering" durability without having to do any freezing and thawing at all. When we are able to reach this stage of development it will be mainly because we shall have come to understand fully the complex mechanism of freezing-and-thawing action in concrete.

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PREPARED BY STANTON WALKER[13]

The National Sand and Gravel Assn. Research Foundation at the University of Maryland has invested most of its time since 1940 in freezing-and-thawing tests of concrete. Whether that investment is to pay off depends on answers to several questions, most of which have been discussed by Mr. Withey. All of these questions have to do with interpreting the test results.

The principal objective of our work has been to evaluate the effects of aggregates of different characteristics on the durability of concrete. To that end a large number of aggregates have been incorporated in concrete subjected to freezing and thawing. Most of these materials have been gravels, although some other coarse aggregates and some sands have also been tested. In general, materials have been selected from sources concerning which something is known of their service record.

Due to limitations of time, this discussion will be restricted to coarse aggregate studies. While procedures have been varied widely in special investigations, our standard test for evaluating coarse aggregates may be described briefly as follows:

1. The coarse aggregate is separated into different sizes and recombined to a selected grading—generally 25 per cent No. 4 to $\frac{3}{8}$ in., 50 per cent $\frac{3}{8}$ to $\frac{3}{4}$ in., and 25 per cent $\frac{3}{4}$ to 1 in.

2. The coarse aggregate, in a room-dry condition, is subjected to a vacuum represented by a pressure of about 1 cm. of mercury for a period of $\frac{1}{2}$ hr. The vacuum is "broken" by the introduction of water, and the aggregate is kept immersed for a period of approximately 24 hr. before being incorporated in the concrete.

3. The concrete used is, conventionally, a well-designed, workable mix of 3 to 4-in. slump and containing 5.5 sacks of cement per cubic yard.

4. The specimens are beams, 3 by 4 by 16 in., with the 3-in. dimension representing the depth as molded.

5. The specimens are cured in a standard moist room for 28 days, followed by 28-day immersion in water at room temperature.

6. Freezing is in air at 0 F. and thawing is in water at 40 F. In order to utilize the freezing-and-thawing units 24 hr. each day, and at the same time perform the work within usual hours, two combinations of freezing and thawing are used: 17 hr. in the freezer and 7 hr. in the thawing tank; or 7 hr. in the freezer and 17 hr. in the thawing tank. The test procedure is started by placing the specimens in the thawing tank. These two combinations are not used interchangeably, independent series of tests being assigned to each.

7. The rate at which the concrete beams reach 0 F. varies with the efficiency of the compressor. When the compressor is operating at good efficiency, about 4 hr. is required. Decreased efficiency has led to periods as long as about 6 hr. This does not quite tell the story. Under efficient operating conditions the air in the ice box reaches 0 F. within about $1\frac{1}{2}$ hr.; under inefficient operating conditions as long as 5 hr. has been required.

8. The results of the freezing-and-thawing tests are evaluated by determining the modulus of elasticity of the specimens by dynamic methods. These determinations are supplemented by flexure tests of the frozen-and-thawed specimens, at the selected end point, compared with flexure tests of companion specimens cured under standard conditions.

9. Modulus of elasticity determinations are made after 1, 2, 3, and 5 cycles,

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[13] Director of Engineering, National Sand and Gravel Assn., Washington, D. C.

after each 5 cycles thereafter through 50 cycles, after each 10 cycles through 200 cycles, and after each 20 cycles thereafter.

10. Freezing and thawing is continued until specimens have suffered a loss of 50 per cent in modulus of elasticity, at which time they are broken in flexure.

The testing procedure described is quite severe. While some groups of specimens withstand failure for several hundred cycles, most failures of aggregates considered good occur between 50 and 150 cycles, and a very large number of failures, for aggregates which have been used with apparent satisfaction for many years, are at less than 50 cycles.

In general, three to five specimens are made for each condition. The results of the dynamic modulus of elasticity, *E*, tests for companion specimens are averaged for each cycle read, so long as all specimens in the group are under tests. When one specimen has been discontinued, because of having suffered a loss of 50 per cent in dynamic *E*, the averaging is stopped, but tests are continued on the remaining specimens. When all specimens in a group have failed, the "average last cycle," and the dynamic *E* for it, are computed. That average last cycle is the arithmetic average of the numbers of the last cycle to which each specimen was subjected. The dynamic *E* for the average last cycle is the arithmetic average of the last determined *E*'s. This last point, with few exceptions, fits well on the curve of points resulting from averages of all specimens at the same number of cycles. The averaging procedure described is carried out with no attempt to discard discordant values, except when specimens or readings are obviously defective.

The results of our tests suggest a discernible relationship to service record, but one so bedevilled with exceptions and erratic results that it cannot be said to be satisfactorily quantitative. However, considerable confidence is had in tests, making direct comparisons between "unknown" aggregates and one of known good service record and good uniformity.

Since I am familiar with difficulties in reproducing results experienced by others, at least as well equipped as ourselves, I do not hesitate to bare the skeleton in our closet. Our "uniformity" difficulties fall in two principal categories:

1. Lack of agreement among companion specimens which had been molded and handled in as nearly the same manner as can be assured by careful workmanship.

2. Lack of agreement among different groups of tests made at different times on the same and similar aggregates.

It is to the difficulties in the first category that we have devoted most study, although we have not neglected those in the second. We have developed some interesting leads toward explanations and have done much speculation, but have arrived at no satisfactory answer.

Mr. Withey has pointed to the importance of the moisture content of the concrete—and he might have added, the distribution of the moisture in the specimens. I reported tests, in a recent unrecorded session at the annual meeting of the American Concrete Inst., showing identical concrete to be broken down under identical freezing-and-thawing procedures, in as few as 3 cycles or to remain intact for more than 150 cycles, depending upon its moisture condition. Accordingly, it seems reasonable that much of the disagreement in test results of supposedly identical specimens, identically treated, may be due to uncontrolled (perhaps uncontrollable) differences in the amount of moisture taken up during the thawing operation.

One group of specimens made with three different crushed stones, each within itself apparently homogeneous, gave results which were very erratic as to the number of cycles required for failure of companion specimens. However, there was close agreement for all specimens between loss in dynamic E and changes in moisture content shown by carefully measured changes in weight. The characteristic action was, first, a slight progressive decrease in weight of the order of less than 0.2 per cent, followed by a gain. By the time the original weights of the specimens were regained the loss in dynamic E was uniformly of the order of 30 to 40 per cent. We felt we were on the right track. Perhaps we are, but the same type of analysis has not held up for other groups of tests. However, in such cases, other significant factors suggest themselves and these studies are being continued.

The small beams used (3 by 4-in. cross-section) may represent a cause of poor uniformity, particularly in the case of nonhomogeneous aggregates. A single unsound piece in one position may do great damage and none in another. Recent tests, where controlled quantities of soft stone and shale were added to hand-picked gravel, lend support to this reasoning. Perhaps our procedures for saturating the aggregates needs reexamination. "Room-dry" may not be a sufficiently uniform starting point for the vacuum saturation process. Experiences with saturating concrete under a vacuum suggest that its moisture condition has a considerable effect on the degree to which the air in the concrete can be removed, and, perhaps, the initial moisture condition of the room-dry aggregate should be examined in the light of that experience. Whether or not aggregates are vacuum saturated, their moisture condition is of significance.

In the second category some lack of agreement among groups of tests made at different times are to be expected and seem to be inherent in concrete testing. However, the disagreements in results of freezing-and-thawing tests which we have experienced are far greater in magnitude than for strength. There can be little doubt but that the particular lot of cement used has a most potent influence. Probably other factors also enter. Whatever the cause, we have had the unpleasant experience of finding it necessary to discard the results of all tests made over a given period of time, because all of the concrete failed so rapidly that suitable comparisons could not be made between unknown aggregates and aggregates of known good performance.

Freezing-and-thawing tests should represent a most useful tool. However, before that usefulness can be fully realized much more must be learned about their conduct and the interpretation of the results.

PREPARED BY FREDERICK O. ANDEREGG[14]

In setting up standard methods of test it is often profitable to make as thorough an analysis as practicable of the factors involved and one is attempted here. The phenomenon of the expansion of water on freezing makes it seem plausible to consider it to be the main cause of disintegration of concrete and other building materials, and the use of freezing and thawing as a test of durability naturally follows. The disruptive forces accompanying freezing of water are, however, modified by a variety of factors, in addition to certain other phenomena which occur with changing temperatures and with variations in moisture conditions. Some of these may have even greater effect than freezing on the results obtained in the field. Some of the factors affecting the concrete integrity are:

1. Water freezing under suitable confinement may produce pressures of the

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[14] Consulting Specialist on Building Materials, John B. Pierce Foundation, Raritan, N. J.

order of 100,000 psi., but the presence of empty pores within the structure often affords an appreciable buffering action against the development of extreme stresses. The correlation of the "saturation coefficient" of bricks with their resistance to freezing and thawing illustrates this point. Therefore, a serious attempt should be made to set up standard conditions for the degree of filling the pores in the concrete specimen. The use of a vacuum, together perhaps with subsequent pressure, should be carefully studied as to the effect on the saturation coefficient and as to the fairness of the subsequent exposure.

The benefit of controlled air entrainment is probably largely derived from this buffering action, and we should be reasonably sure that any testing procedure will not fill these specially added pores to any greater extent than they would be filled under natural exposure.

2. The speed of temperature reduction is a factor contributing to the manner of growth of the ice crystals. If it is very slow, large dagger-like crystals several inches in length may be formed, as under the leaf mold in the woods in the winter, to produce forces in certain directions. The lowering of the melting point of ice, however, under such ice crystals, prevents this unidirectional growth so that this effect may probably be safely disregarded.

3. Rapid temperature reduction may set up other harmful forces because of different coefficients of thermal expansion found among the constituents of concrete. Portland cement has a thermal coefficient dependent upon the composition, the age, and the state of hydration and subsequent dehydration which is comparatively high among the inorganic materials usually found in concrete. Calcareous aggregates have a lower coefficient than siliceous aggregates, and a combination of, say, marble with white cement at the age of about one year is subject to rather large differential straining with rapid temperature changes. Again the surface of the eastern face of a tall building has to resist spalling forces caused by rapid heating up by the morning sun and the western face by the rapid cooling of a thunderstorm in the evening. Therefore, the size of the specimen should be standardized as well as the rate of cooling and warming. In evaluating the results, cognizance should be taken of the thermal expansion of the constituents and also of possible resilient factors. Incidentally, it is respectfully suggested that more attention to increasing such factors might well improve concrete durability.

4. A very important factor in the durability of concrete is the presence of salts. The sulfates and chlorides of calcium, magnesium, and sodium are apt to give most trouble and in several ways. In the first place their interaction with calcium aluminate to form double salts with volume expansion is a cause of obvious worry; but it seems probable that greater harmful effect is often produced by oriented crystal growth. As temperature is slowly lowered in the concrete, opportunity is provided for the crystal growth of many salts predominantly along one axis, and the forces involved in such crystal growth are of the order of 100,000 psi. or more. A million self-acting jacks may be growing in the concrete. The writer has noted that concrete disintegrates, when saturated with strong sodium sulfate solution, the faster the slower the rate of cooling. It is suggested that by so controlling the cooling rate of a specimen immersed in sodium sulfate solution as to take a week to go from, say, 160 to 0 F., a good estimate would be obtained in the one experiment of the ability of the concrete to withstand such internal expansive forces.

5. In the actual exposure of most concrete, variable moisture conditions are encountered, which may produce a variety of effects: (*a*) Detrimental sulfuric acid of winter rains or soluble salts

may be carried in under certain conditions, (*b*) Again a beneficial leaching action may be brought about whereby deleterious salts are removed. Under such conditions some of the lime of the calcium hydrosilicate binding gel may also be removed with more or less unfortunate effect on the integrity of the cement, (*c*) With moisture changes the growth of the oriented crystals is often encountered and a tendency for them to accumulate in the pores immediately behind the surface has been noted. Any surface skin or crust is often pushed off by the developing crystals. (*d*) Moisture in the concrete promotes autogenous healing so that the harmful effects of the various disintegrating forces are very often more or less repaired, given moist conditions and time.

RECOMMENDATIONS FOR TEST SPECIFICATIONS

The following should be accurately controlled:

1. Size and shape of concrete specimen,
2. Saturation coefficient,
3. Rate and extent of cooling, and
4. Rate and upper temperature of thawing.

If it is desired to determine chiefly the effect of differences in thermal coefficients of expansion, rapid changes in temperature up and down between, say, 30 and 160 F. should be brought about.

If it is desired to determine quickly the resistance of the concrete structure to internal pressure brought about by ice alone the saturation coefficient should approximate 100 per cent.

To get an estimate of the ability of the concrete structure to resist the expanding action of oriented salt crystals, saturate at about 160 F. in a 20 per cent sodium sulfate solution in a container. Provide the container with sufficient insulation so that it requires about one week for cooling.

PREPARED BY GUY H. LARSON[15]

The purpose of the State Highway Commission of Wisconsin in making freezing-and-thawing tests on concrete has been to compare the durability of concrete containing certain unknown or doubtful coarse aggregates with that of concrete containing aggregate of known satisfactory quality, and to determine the effect on the durability of concrete of various cements and admixtures. The test used—specimens partly immersed in water for both freezing and thawing—would be classed as type 2 according to Mr. Withey's discussion of freezing-and-thawing tests.

Our facilities consist of a "walk-in" freezing room $8\frac{1}{2}$ by $12\frac{1}{2}$ ft., with 8 in. of cork insulation. The refrigerating system is of the ammonia expansion type. The condenser is a self-contained unit of 4.5 tons capacity when operating at 5 F. evaporating temperature and 86 F. condensing temperature. The evaporator or freezing coils consist of 1800 ft. of $\frac{3}{4}$-in. galvanized pipe supported near the ceiling above an insulated deck. Two blowers circulate the air through the coils and the refrigerated space below. The freezing room is maintained at a temperature of −10 F. There is a $7\frac{1}{2}$ by $8\frac{1}{2}$-ft. anteroom with 5 in. of cork insulation, which is maintained at a temperature of 40 F.

For convenience in moving specimens in and out of the freezer, they are placed in metal trays (approximately 25 by 37 by $6\frac{1}{2}$-in.) which are loaded one above the other with a minimum clearance of 5 in., in racks mounted on four-wheeled hand trucks.

The concrete specimens generally used are 3 by 4 by 16-in. or 6 by 6 by 20-in. beams, depending upon the maximum size of the aggregate. Specimens are made in accordance with standard procedure. They remain in the molds in the mixing room for 1 day, after which they are stripped and cured in the moist room for 26 days, and then submerged in water for 1 day. At the end of the 1-day soaking period, the specimens are placed in the trays on the trucks with a minimum distance between specimens of 1 in. The trays are filled with water to a depth of $\frac{1}{2}$ to $\frac{3}{4}$ in. and the loaded trucks pulled into the freezing room. After 18 hr. the trucks are removed from the freezer and the specimens thawed for 6 hr. at room temperature. Specimens remain in the trays on the trucks throughout the freezing-and-thawing cycle. A large fan is so placed as to blow the chilled air away from around the bottoms of the trucks. Without this precaution the lower tray usually is not entirely free of ice at the end of the 6 hr. After the thawing period, the water in the trays is brought to the proper depth again, the loaded trucks pulled into the freezer, and the next cycle started.

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[15] Senior Assistant Engineer, State Highway Commission of Wisconsin, Madison, Wis.

The rate of freezing is slightly different at different points in the freezing room, and the rate of freezing and thawing is slightly different in each of the trays on a truck. In order to offset this the trucks are rotated in position in the freezer each cycle, as are also the three trays on a truck.

When operating with full load, the concrete, after the first cycle, enters the freezer at an internal temperature of approximately 63 F., is lowered to 32 F. in approximately 1 hr. and 5 min., and to -8.5 F. in approximately 8 hr. When removed from the freezer, the concrete is at practically -10 F., is raised to 32 F. in 1 hr. and 40 min., and to approximately 63 F. at the end of the thawing period.

When comparing different aggregates or cements, the freezing-and-thawing cycles are usually continued until the specimens show some sign of damage. They are then all removed from the freezer, brought to room temperature, and tested for strength. When it is desired to have more accurate information on exactly what is happening to the concrete as the test progresses, determinations of the dynamic modulus of elasticity of the specimens are made at certain intervals by means of sonic apparatus available to us at the University of Wisconsin. Companion specimens, cured continuously in the moist room, are always made for reference.

One serious objection to our present freezing-and-thawing test is the length of time it requires. The action can be greatly accelerated by forcing more water into the concrete, but there are objections to this procedure also. Even very satisfactory concrete can be made to fail rapidly in this way, but the differentiation between durable and nondurable concrete usually is adversely affected.

We are planning to try freezing the specimens from one side, with water present at the opposite side. An insulated tank with open top, to be placed in the freezing room, has been built for the purpose, but so far we have been unable to obtain the necessary heating elements and controls. The proposed procedure is to fill the tank partially with completely saturated sand on which the specimens will rest with their upper surfaces exposed to the low temperature in the freezer. The space between and around specimens is to be sealed with some insulating material. The lower surfaces will be kept at some temperature above freezing by means of the heating elements in the sand below. The idea is to retard, or perhaps prevent, the movement of the freezing "isotherm" entirely through the concrete, thereby providing more time for water to move into the concrete under conditions and forces at least simulating those it will encounter in service.

GENERAL DISCUSSION

MR. WALTER H. PRICE.[1]—I should like to ask Mr. Valore how he extracts the air from the rubber tube containing the test specimen in the procedure he described? I am interested because the failure of a specimen in three cycles of freezing and thawing, as mentioned by Mr. Walker, can be accomplished by extracting air from the specimen and saturating the specimen under a vacuum before starting the cycles of freezing and thawing.

MR. RUDOLPH C. VALORE.[2]—We have ordinarily had these bags made of rather thin ($\frac{1}{16}$-in.), flexible rubber, of inner tube quality. When a bag containing a concrete specimen and about one pint of water is immersed in water to within a few inches of the open top end, the external pressure is usually sufficient to expell air entrapped between the bag and the contained specimen. Any remaining air may be forced out by manipulation before the bag is sealed shut. The purpose in removing this air is to provide for complete immersion of the specimen in the water within the bag, but not to remove air from the specimen. I do not believe that there is any relation between this operation and that used by Mr. Walker, which resulted in three-cycle failures.

We are fairly certain that the air is removed from the bag since, upon removing a frozen specimen from the bag, we find it completely coated with ice.

MR. M. O. WITHEY.[3]—I should like to add some information about the effect of long curing under water, and thereby thorough saturation of the specimen. In the tests made by the Highway Research Board Committee, concrete of good aggregate, as well as concrete of a poor aggregate was made at the Public Roads Administration Laboratory, and specimens from each of those concretes were delivered to each of the coordinating laboratories. One of the coordinating laboratories (A), in its runs on these two types of concrete, had quite diverse results from those obtained by other laboratories. Since we had some extra specimens which had been made and then cured in water for a period of two years, at the P.R.A. Laboratory, we decided to repeat the tests at Laboratory A. Hence we sent part of those extra specimens to Laboratory A and part to Laboratory B, at which an average number of cycles for failure had been taken in the original program. On the specimens made with good aggregate, Laboratory A reported 167 cycles to produce failure after 28 days of water curing, but only two cycles after the specimens were soaked two years. On the specimens made of poor concrete, Laboratory A reported 27 cycles to disrupt the concrete after curing 28 days, but only two cycles after curing two years. At Laboratory B 27 cycles were required to disrupt the good concrete after curing 28 days, but only four cycles were required after curing two years. The number of cycles was four on the poor concrete after curing twenty-eight days, and after curing two years the number of cycles was two. The main conclusion to be drawn is that the two-year-old concrete of both good and poor aggregate, was *thoroughly saturated* with water and required only a few cycles of freezing and thawing to cause failure. One will find the same thing is true if specimens, which have been subjected to 600 or 700 cycles of freezing and thawing without signs of breakdown, are then submitted to a vacuum, filled with water, and then frozen and thawed. Specimens so treated will break down in two or three cycles. Hence the quantity of water which is put into the pores of hardened concrete has a very vital effect on its durability under any freezing-and-thawing program.

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[1] Materials Engineer, U. S. Bureau of Reclamation, Denver, Colo.

[2] Materials Engineer, National Bureau of Standards, Washington, D. C.

[3] Professor of Mechanics, University of Wisconsin, Madison, Wis.

I should like to say one thing more about the loss of weight. Loss of weight has very little significance for concrete cured in air and frozen in air. It has some significance when concrete is frozen in water, but a good strong concrete having a strength of 3500 to 5000 psi. at 28 days, when subjected to freezing and thawing a sufficient length of time to reduce the weight 25 per cent, would have practically no crushing strength and no transverse strength. Therefore, in the Highway Research Board program, it was decided to use the transverse bending test, which is a much more sensitive index of loss in strength than is the loss in weight.

MR. PRICE.—Were those old specimens Mr. Withey just described as having failed in a few cycles, frozen in water-filled metal containers? I ask, because in such containers the ice first forms around the specimens at the ends and the specimens may be ruptured in tension by the expanding forces exerted against those end ice grips.

MR. WITHEY.—In answer to Mr. Price's question, it should be noted that the specimens tested in the Highway Research Board coordinating program were all frozen in air. We have had the same experience which he reports when specimens have been immersed in water with their bottoms in contact with a metal tray. In both the coordinating program and in programs involving immersion of the specimen in water during freezing, we have supported the specimens on thin strips of wood.

MR. K. B. WOODS.[4]—I should like to supplement Mr. Walker's comments in regard to the moisture content at the time of freezing and thawing. We have been carrying on a very active program for a number of years in connection with service of pavements, and now in recent months have been endeavoring to make concretes and trying to get accelerated testing procedures developed which will give us results in a few cycles that are comparable with the field exposures after 10 or 15 yr. We find that we get a much lower saturation coefficient for our good aggregate than we get for our poor aggregate. Likewise, we can obtain failure at a very low number of cycles when we vacuum-saturate our poor aggregate; and in contrast, our good aggregates all go many cycles before they fail, even though vacuum-saturated.

MR. IRA PAUL.[5]—The one thing we have to contend with in New York State is not so much the action of freezing and thawing of water on concrete as the effect of the application of salt and calcium chloride for ice control on concrete pavements. Many of our concrete roads were scaling and disintegrating because of the adverse effect of chloride salt action. Several years ago an investigation was initiated by our laboratory to try to do something about this. In a series of laboratory freezing-and-thawing tests exposed to the action of chloride solutions it was discovered that a certain type of admixture when incorporated in the concrete mix would resist the destructive action of these salts. Subsequently a three-lane concrete pavement on a heavy grade was set up as a field experimental project to check on laboratory findings. Portland cements of various compositions were used, and the admixture was incorporated in alternate slabs all along the three-lane highway. When the project was completed cores were removed from all sections of the pavement and subjected to freezing-and-thawing tests in a 10 per cent calcium-chloride solution. Briefly summarized, the laboratory freezing-and-thawing tests in the chloride solution showed that practically all the straight portland-cement concrete cores scaled and disintegrated with quite heavy losses between 25 and 50 cycles, whereas the portland-cement concrete with the admixture showed much better resistance to chloride action. A field examination of the pavement was made the following spring after it was constructed. During the winter season there were many applications of chloride salt with the abrasive for ice control on the heavy grades. Where chloride salts were applied, the slabs containing the straight portland cement had scaled badly regardless of the composition of the cement, whereas the concrete slabs containing the admixture were in excellent condition. Thus it was quite interesting to observe the excellent correlation between the laboratory freezing-and-thawing tests on the cores removed from the pavement and the field service tests.

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[4] Associate Director, Joint Highway Research Project, Engineering Experiment Station, Purdue University, Lafayette, Ind.

[5] Director, Public Works Lab., New York State Department of Public Works, Division of Construction, Albany, N. Y.

Some years ago a few concrete cores were removed from a 24-yr. old pavement in good condition and subjected to freezing and thawing in plain water. These cores completely disintegrated between 25 and 50 cycles of freezing and thawing, in spite of the fact that the pavement concrete was in good serviceable condition for nearly a quarter of a century. Just how to interpret the freezing-and-thawing laboratory tests in water is anybody's guess, but we do know from our vast experience over fifteen years that the laboratory freezing-and-thawing tests in chloride solution on cores removed from the pavement do check the field service records where pavements are treated with chloride salts for ice control.

MR. J. C. PEARSON.[6]—A number of people here will doubtless recall a meeting held in Washington some years ago to organize a comprehensive joint investigation of this matter of durability and freezing-and-thawing tests. At that time our technical staff was contemplating an exposure of a series of small concrete beams to the weather, the plan being to have the beams half submerged in flat concrete trays of water on the roof of a storage building adjoining our laboratory in Allentown. Our purpose primarily was to try this out as a natural but severe weathering test, from which significant results might be secured within a few years. I therefore asked the group what might be expected from a series of exposures of this sort, and while different opinions were expressed by different individuals, the general reaction was that such a program would be worth trying, and we accordingly started the tests in the fall of 1941.

During the war years we were barely able to keep up the observations on these specimens, but we did manage to keep the beams in water during the summers, and in water, ice, and snow during the winters, and to make sonic modulus determinations in the spring of each year. We had hope that a 5-yr. exposure, with some 40 or 50 freezings and thawings each winter, would distinguish between the sheep and the goats, perhaps emphasize some of the factors essential to durability, and also show some marked differences in the freezing-and-thawing resistance of different cements.

Without going into much detail, the program included concretes made with fourteen different cements, seven of these comprising the five different types conforming to Standard Specifications for Portland Cement (C 150-44)[7] and the two air-entraining cements, types IA and IIA, conforming to Tentative Specifications for Air-Entraining Portland Cement for Concrete Pavements (C 175-44 T)[8]; the other seven were selected for variations in composition and source. These fourteen cements were the main variable in the test; the seven regular types were made up with cement factors of $4\frac{1}{2}$ and 6 bags per cubic yard of concrete, and the others were used in concretes having slumps of 6, 3, and 0 in. at the same water-cement ratio.

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[6] Director of Research, Lehigh Portland Cement Co., Allentown, Pa.

The present indications from these tests, after 5-yr. exposure, are as follows:

1. The continuous exposure to the weather of the half-submerged beams appears to be less severe than most laboratory freezing-and-thawing tests involving 50 or more cycles. This may be due in part to the fact that the beams are probably not completely frozen in the majority of freezing cycles, and in part to the complete absence of "thermal shock."

2. The 5-yr. plan as originally conceived appears to be quite inadequate for developing any significant differences in the freezing-and-thawing resistance of the 14 cements included in this investigation. None of them has shown any consistent weakness thus far.

3. The group of beams placed by vibration at 0-in. slump has suffered relatively more than any other. This is believed to be due to the use of too stiff concrete and excessive vibration required to consolidate the concrete.

The fact that there has been relatively little difference in the damage suffered by the different types of cement seems to be at odds with the experience reported by a number of the speakers, and suggests that there may be something more than type and calculated composition of cements that determines the resistance of concrete to freezing and thawing.

MR. PRICE.—I should like to make a remark concerning the curves shown in Fig. 11 of my discussion. Concretes in a parapet wall made with the five cements represented in the curves of this figure show little difference in weathering resistance after 3-yr. exposure in the mountains of Colorado.

MR. BAILEY TREMPER.[9]—Our experience indicates that differences in resistance among cements as shown by laboratory tests are often reflected under severe outdoor exposure. This applies to cements combined with alkali-reactive aggregates and also with some aggregates that are not reactive as the term is usually understood. This statement is based in part on the observed behavior of actual structures and in part upon tests of laboratory-made specimens that have been exposed with one face lying on natural ground in a severe climate.

MR. MYRON A. SWAYZE.[10]—I have just two points I should like to bring up. We have been making freezing-and-thawing tests in our laboratory for the past twelve years. During that time we have frozen and thawed several thousand specimens. Practically always we get a softening up of an outside edge with a fairly hard core in the middle, and that will gradually work towards the center. We have rare cases where specimens have been in fairly good external condition, and then in the space of ten cycles have broken down all the way through, like the "wonderful one-hoss shay."

The first question I want to ask is

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[7] 1944 Book of A.S.T.M. Standards, Part II, p. 1.

[8] 1944 Book of A.S.T.M. Standards, Part II, p. 1203.

[9] Materials Engineer, State of Washington, Department of Highways, Olympia, Wash.

[10] Director of Research, Lone Star Cement Corp., Hudson, N. Y.

directed to those who like the sonic methods of test: If you take a specimen in which you have a softened exterior and a perfectly sound core, how can you take a disuniform specimen of that sort and get the true sonic modulus?

The other point in which I have been quite interested is the type of container used for freezing and thawing, and just how the specimen is set so that the only deleterious action the cycle produces is actual freezing and thawing. Mr. Davis showed a circular can. I did not understand whether he said he had water in the can, or cylinder, or whether the specimens inside of the can were in air. If the cylinders were immersed in water, I should like to call attention to the experience we had when we first started freezing-and-thawing tests. We had 3 by 6-in. specimens, and the cans in which we immersed them were galvanized iron 6¼ in. square. We stacked the specimens lying on their sides, one above the other, and subjected them to two cycles of freezing on the first day. Then we looked at them and every one of our specimens was beautifully cracked either in half or in quarters. The reason for this was that, taking a square can and putting the specimens in laid flat, there was a horizontal water space between the blocks, and the blocks first froze to the can all the way around. The water between the blocks was still liquid, and it had no place to go when further freezing occurred. It could not get by the specimens frozen to the edge of the cans, so it promptly went up through the center of the specimens, breaking them as it went. Now, a 3 by 6-in. cylinder will have more resistance than a 6 by 6-in. block 3 in. thick, but you can do a good deal of damage to a specimen of that sort just by the expansion of ice on the exterior of the block. It is, therefore, very important to let the freezing be gradual in one direction. The method we have now is to put one dummy block in the bottom of a can and the test blocks are set in pairs, so that there is a continuous unobstructed water channel from the top to bottom of the can, through the center. This is always the last part of the can to freeze and the free water merely rises as freezing progresses and the specimens are under no external stress at any time during the freezing. That is very important. If you get such strains in your specimens, you are very apt to have a disintegration that is not due to freezing in the block itself. It is just an outside influence.

MR. WITHEY.—In the Highway Research Board program of tests, disintegration over the entire outside of the specimen did not occur since the limiting loss in the dynamic modulus of the elasticity occurred prior to such general failure of the surface of the specimen. We simply made notes from time to time as the tests progressed of the spalling of corners, effects, if any, of unsound aggregate particles and the condition of cracks, if any, and took our dynamic E readings. In such tests it is not necessary to consider the minute changes in dimension in computing dynamic E. I have not had experience with the deterioration which accompanies a 50 per cent reduction in dynamic E, but after a reduction of 30 per cent, the weight loss is less than 1 per cent. Hence the disintegration on the surfaces of the specimens will have very little effect on the dimensions of the specimens.

MR. SWAYZE.—If you take a composite bar with a core of high-strength concrete and surround it with, let us say, a layer of low strength plaster of Paris, and then take a sonic modulus reading, would you not get the sonic modulus of the plaster rather than that of the inside core of hard concrete?

MR. WITHEY.—No, you would get dynamic E for the composite beam.

We have not considered it desirable in the light of our experiences to place specimens on top of one another in a vertical container in either of the Highway Research Board coordinating or local programs. We have separated the specimens and supported them on shelves or racks in separated layers.

MR. F. B. HORNIBROOK.[11]—Mr. Davis mentioned that he got fairly good correlation between the change in length and the loss in weight of his specimens, but not with the change in dynamic modulus. My experience, however, when testing air-entrained concrete has been that a better relationship between change in length and change in dynamic *E* was obtained than between change in length and loss in weight. It is my understanding, however, that most of the work Mr. Davis is reporting was done with concretes not containing air-entraining agents. Along this same line, I should like to ask Mr. Price whether they measure change in weight and, if so, whether the changes in weight bear an apparent relationship to change in length or to change in dynamic modulus?

MR. PRICE.—We have taken sonic modulus readings in conjunction with many freezing-and-thawing tests. Our tests have shown a rapid reduction in sonic modulus for concretes of poor quality; but it is not uncommon to obtain an increase in sonic modulus for a considerable number of freezing-and-thawing cycles for the better concretes. Good correlation is obtained between changes in sonic modulus and expansion of the concrete. At present we prefer expansion measurements as an early indication of failure.

We are now considering a bar type specimen on which expansion, sonic modulus, and weight loss may be obtained. At failure there is good agreement among these three measurements, but in the early stages of the tests the expansion and weight loss may remain practically constant while the sonic modulus is showing a slight increase.

MR. WITHEY.—I wonder whether the good concretes mentioned by Mr. Price are not having their early strengths increased by the process he uses for thawing in water.

MR. PRICE.—For some of the concretes of good quality the increased hydration during the early stages of the test, caused by the warm thawing water, does appear to more than offset any deterioration due to freezing and thawing. A more severe cycle would show less net gain in strength.

Some years ago we subjected specimens to cycles of freezing and thawing, and wetting and drying periods. In some cases, the drying cycle materially improved the resistance of the concrete to freezing and thawing.

Mr. Valore mentioned that a correlation of permeability of the concrete with the freezing-and-thawing results might furnish us with valuable information on the expected behavior of the concrete. Our tests show good correlation between permeability and resistance of the concrete to freezing and thawing for concretes made without air-entraining agents and a fair correlation for those containing air-entraining agents. Recent tests made at high water pressures of 400 psi. on concretes containing various amounts of air up to 25 per cent show a slight increase in permeability up to about 3 per cent air and a decrease in permeability from there up to about 10 per cent air content where the permeability again began to increase. The durability of the same concretes increased with increased air content up to at least 8 per cent air.

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[11] Materials Engineer, National Bureau of Standards, Washington, D. C.

MR. STANTON WALKER.[12]—With reference to Mr. Price's comments that good concrete increases in modulus of elasticity as freezing and thawing progresses, I suggest that he examine the moisture condition of his specimens. If the indications of our own experiences are accurate, it will be found that these increases in modulus of elasticity are accompanied by gains in weight of the specimens. It is believed that the absorbed water, indicated by the gain in weight, is responsible for the increased modulus of elasticity.

In our standard procedure, for which the specimens are thoroughly saturated before freezing and thawing is started (see my prepared discussion), there is always a sharp, though relatively small, decline in modulus of elasticity during the first few cycles of freezing and thawing, accompanied by a decline in weight. In certain of our tests, where the concrete was not thoroughly saturated, we have found an increase in modulus of elasticity accompanied by a gain in weight.

That moisture condition affects the modulus of elasticity of concrete measured dynamically is well illustrated by many data. Without attempting to recall all sources of such information' reference is made to the paper by Obert and Duvall[13] presented before the Society in 1941 and to my paper[14] on "Resistance of Concrete to Freezing and Thawing as Affected by Aggregates" published by the National Sand and Gravel Association.

MR. PRICE.—It is agreed that the condition of saturation of the concrete has a marked effect on the number of cycles of freezing and thawing it will withstand. However, the test is designed to give some indication of the resistance of the concrete to weathering and not to fail the specimen as rapidly as possible. We know that we can fail fully saturated air-entrained concrete in a few cycles, yet partially saturated air-entrained concrete performs very well under severe weathering conditions. In general, in our tests fog-cured specimens are soaked for 24 hr. and dry specimens are soaked for 96 hr. before starting the freezing-and-thawing cycles. The specimens are kept under water during the entire period of tests except for the short periods when they are measured for weight loss and expansion.

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[12] Director of Engineering, National Sand and Gravel Assn., Washington, D. C.

[13] Leonard Obert and Wilbur I. Duvall, "Discussion of Dynamic Methods of Testing Concrete with Suggestions for Standardization," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 1053 (1941).

[14] Stanton Walker, "Resistance of Concrete to Freezing and Thawing as Affected by Aggregates," Nat. Sand and Gravel Assn., Circular No. 26.

THE INFLUENCE OF GYPSUM ON THE HYDRATION AND PROPERTIES OF PORTLAND CEMENT PASTES[*]

By William Lerch[1]

Synopsis

The manner in which the added gypsum controls the setting of cement has been the subject of many investigations and of some controversy. It is generally agreed, however, that its effect is to stop the rapid reaction normally shown by the calcium aluminates. In the absence of precise information relative to the function of gypsum in regulating the initial hydration and in order to provide ample protection against the abnormal expansion that might result from the use of excess amounts of gypsum it has been the custom from the beginning to place a limit on the SO_3 content of portland cement in standard specifications. In the A.S.T.M. specification for cement this limit has not been changed since 1917[2] except to permit a higher value for high early strength cement. In the belief that the quantity of gypsum required to regulate properly the hydration and hardening would vary with cement composition and fineness, the studies described in this paper were undertaken.

Twelve commercial clinkers covering the range of chemical composition generally encountered in portland cements were selected for study. Each of these compositions was used with various SO_3 contents with fineness held uniform at about 1900 sq. cm. per gram. In addition five of the clinkers were ground to varying degrees of fineness with SO_3 constant at about 1.8 per cent.

The effect of SO_3 on the rate of hydration was measured with a conduction calorimeter on neat cement pastes. Mortar prisms were used for physical tests. These studies have shown that a proper regulation of the reaction of early hardening is of much greater importance than merely regulating the time of setting. Such regulation not only influences the rate of hydration, but affects the strength and the volume-change characteristics of the hardened cement paste. With some cement compositions the gypsum retards the initial hydration and set, while with others it acts as an accelerator.

For many cements the strengths can be increased and the contraction on drying decreased by the use of larger additions of gypsum than are permitted by current specifications. The quantity required to obtain the best strength and the least contraction can be used without danger of abnormal expansion in water storage. The results show that the fineness, the alkalies, and the $3CaO \cdot Al_2O_3$ content all influence the gypsum requirements.

The paper contains a discussion of the function of gypsum in regulating the reactions of hydration and hardening.

When portland cement clinker is ground without the addition of a retarder, the reactions with water are usually so rapid that quick set occurs. Of the phases normally present in portland cement clinker, the alumina-containing phases appear to be those which react with sufficient rapidity to give rise to the undesirable quick set. Since alumina-containing phases are always present, it is the common practice in the manufacture of portland cement to add some material to control the rate of the initial reactions. At present, gypsum is the retarder generally used. The manner in which the added gypsum controls the setting of cement has been the subject of many investigations and of much controversy. It is generally agreed, however, that its effect is to stop the rapid reaction normally shown by the aluminate phases, and that it causes the formation of calcium sulfoaluminate in place of hydrated calcium aluminates.

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[*] Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] Research Chemist, Research Laboratory of the Portland Cement Assn., Chicago, Ill.

[2] Standard Specifications for Portland Cement (C 9 – 17), 1916 Book of A.S.T.M. Standards, p. 429.

The problem of retarders and accelerators for portland cement is of importance, not alone for the effect which they may have upon the rate of hardening, but also for their effect upon the structure developed by the paste at early ages and the manner in which this structure may alter the physical properties of the hardened paste at subsequent ages.

It seems apparent that the fineness of the cement would be an important consideration in determining the quantity of gypsum required to obtain proper retardation. However, bodies responsible for cement specifications do not seem to have taken this into account. For example, the Standard Specifications for Portland Cement adopted by the A.S.T.M. effective January 1, 1917, (C 9 – 17)[2] limited the SO_3 content to 2.0 per cent and permitted a residue of 22 per cent on the No. 200 sieve. Since that date there has been a progressive increase in the fineness of portland cement, and in the most recent A.S.T.M. Specifications (C 150 – 44)[3] a minimum limit of fineness has been imposed of 1600 sq. cm. per g. specific surface (**1**).[4] This limit would be equivalent to approximately a 5.0 per cent residue on the No. 200 sieve. Despite this large change in fineness there has been no change in the specifications to permit larger additions of gypsum to compensate for the increase in fineness—except that for high early strength cement an SO_3 content of 2.5 per cent is permitted.

The present investigation was undertaken to examine the influence of gypsum on the hydration and properties of portland-cement pastes when using cements of different composition and with fineness varied. Twelve commercial clinkers, representing the range in chemical composition encountered in portland cements, were used in the investigation. The clinkers with various additions of gypsum were ground to the same specific surface and the rate of heat liberation was determined for the cements. The cements were also used in the preparation of mortar specimens to determine the effect of the various additions of gypsum on the physical properties of the hardened paste. For five of the clinkers additional tests of the rate of heat liberation were made in which the specific surface was varied while holding the SO_3 constant. The discussion of the data contains a description of the nature of the reactions of gypsum with portland cement, and the manner in which gypsum affects the rate of heat liberation and the physical properties of the hardened paste.

APPARATUS AND METHODS OF TEST

The Conduction Calorimeter:

In previous investigations of retarders for portland cement the work has often been confined to a determination of the effect of retarders on the time of set by the conventional penetration tests. In the present investigation, the rate of hydration at early ages was adopted as a means of studying the effect of gypsum. For this purpose the rate of heat libera-

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[3] Standard Specifications for Portland Cement (C 150 – 44), 1944 Book of A.S.T.M. Standards, Part II, p. 1.

[4] Boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1292.

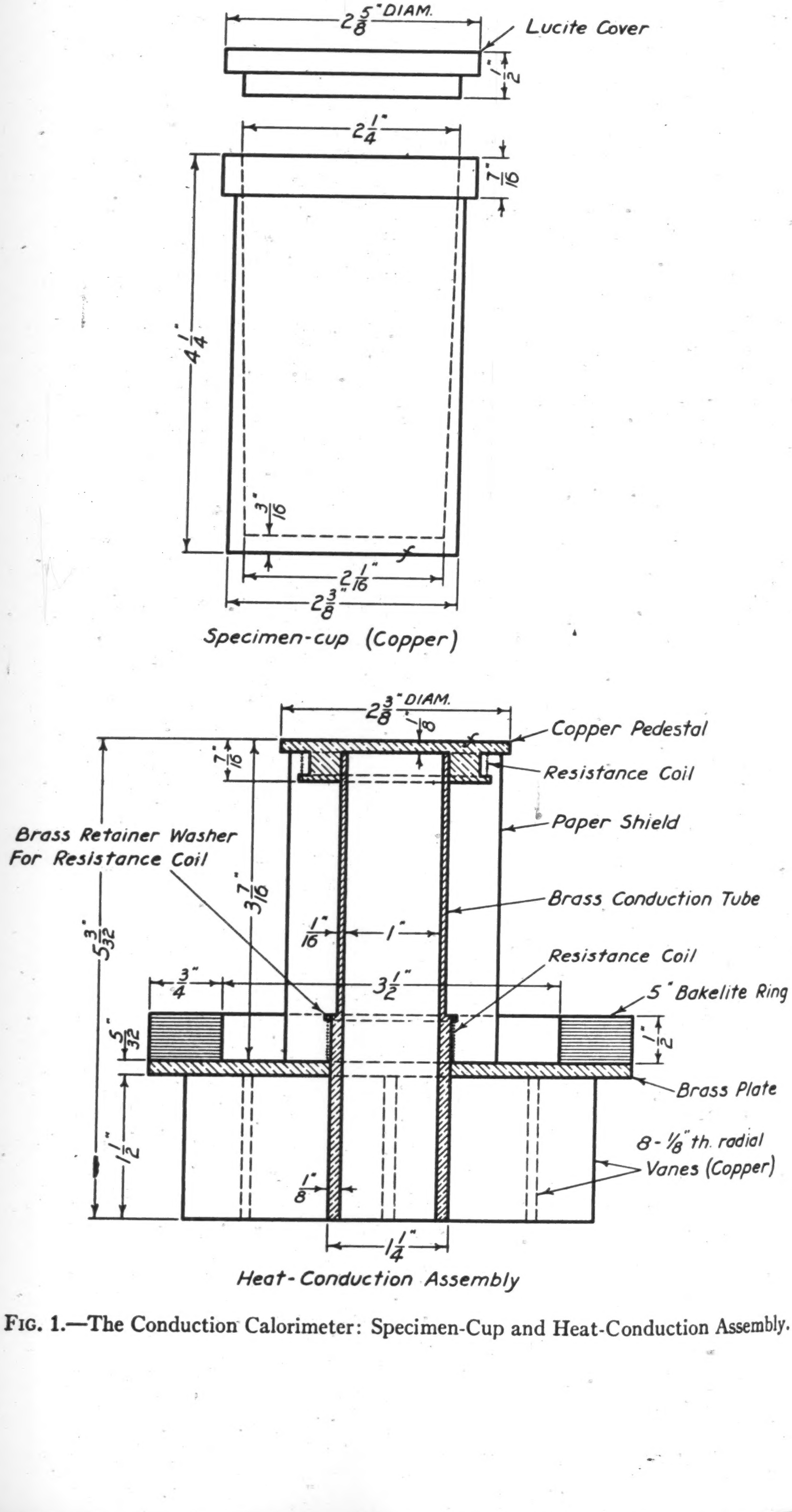
2 5/8" DIAM.
Lucite Cover
1/2"
2 1/4"
7/16"
4 1/4"
3/16"
2 1/16"
2 3/8"
Specimen-cup (Copper)
2 3/8" DIAM.
1/8"
Copper Pedestal
7/16"
Resistance Coil
Paper Shield
Brass Retainer Washer For Resistance Coil
3 7/16"
5 3/32"
Brass Conduction Tube
1/16"
1"
Resistance Coil
3/4"
3 1/2"
5" Bakelite Ring
5/32"
1/2"
Brass Plate
1 1/2"
8 - 1/8" th. radial Vanes (Copper)
1/8"
1 1/4"
Heat-Conduction Assembly

FIG. 1.—The Conduction Calorimeter: Specimen-Cup and Heat-Conduction Assembly.

tion was used as a means of measuring the rate of hydration. The calorimetric methods were similar to those used by Carlson (2) and Forbrich (3).

A conduction calorimeter designed according to recommendations of R. W. Carlson[5] was used for the determination of the rate of heat liberation during the first 72 hr. The calorimeter proper consists of four essential parts which are shown in Figs. 1 and 2: (1) a copper cup to contain the cement paste, (2) a brass tube to conduct the heat away from the specimen cup, (3) resistance thermometers intimately secured to the two ends of the conduction tube, and (4) a heat-dispersing base immersed in a water bath. When the calorimeter is in operation, the specimen cup, containing neat paste (water-cement ratio = 0.40 by weight), is placed on the copper pedestal and a vacuum flask is placed over the apparatus to minimize loss by radiation and by air-convection. Thus virtually all the heat generated by the hydration of the cement in the specimen cup is conducted to the water bath. The complete assembly of six units is shown in Fig. 3.

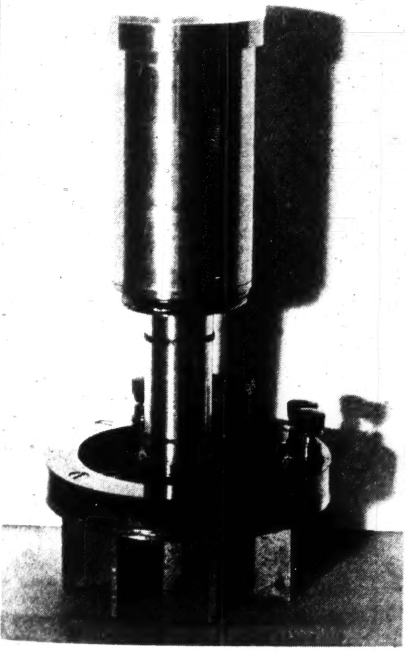

FIG. 2.—One of the Heat Conduction Units.

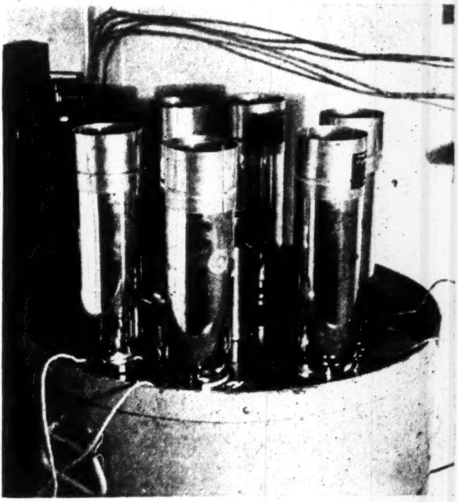

FIG. 3. Assembly of Six Units in Operation.

The rate at which heat is conducted away from the specimen is proportional to the difference in temperature between the specimen cup and the water bath. The difference in temperature is measured by means of a Leeds and Northrup instrument which records the difference between the temperatures (in terms of resistance) at the ends of the conduction tube. The instrument records the resistance difference for each of six calorimetera at 6-min. intervals. A detailed description of the method of calibration, and computation of data are given in a previous paper by Carlson (4).

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[5] Personal communication.

The Temperature Rise in the Cement Pastes:

Among the cements used in this investigation the temperature rise varied over a wide range; the maximum was 23 F.; the minimum was 4 F. Carlson (2) has shown that in 10-in. slabs with one surface insulated and the other maintained at a uniform temperature, the temperature rise in concrete containing 1.5 bbl. of cement per cu. yd. would range from 10.5 F. for a low-heat cement to 32 F. for a high early strength cement. Thus, the temperature rise in the conduction calorimeter for the neat cement pastes used in these tests is of the same order as the temperature rise in relatively thin concrete slabs and no undue acceleration of the rate of hydration results from the temperature rise in the calorimeter specimen.

Determination of Heat of Immediate Hydration:

Since cement liberates an appreciable amount of heat immediately upon being mixed with water, the heat liberated before the first measurement with the conduction calorimeter must be determined by an independent method. The heat liberated during this initial period is referred to as the heat of immediate hydration. It is determined with a simple bottle calorimeter. The bottle used for these determinations has a capacity of about 300 ml. Three hundred grams of cement and 120 ml. of water are used for the determination. The initial temperature of the water is adjusted in such a manner that the paste at the completion of a determination will be approximately at room temperature. The measured quantity of water is placed in the bottle and the initial temperatures of the water and cement are recorded. The cement is then poured into the bottle, the stopper and thermometer inserted and the contents shaken vigorously for 5 min. Readings of the paste temperature are taken at 5-min. intervals for 30 min. The heat of hydration is then calculated from the temperature rise and the heat capacity of the system (cement, water, and bottle) and designated the heat of immediate hydration.

SELECTION AND PREPARATION OF THE CEMENTS

Twelve commercial clinkers were selected to represent the range of chemical composition found in portland cements. The chemical analysis and calculated compound composition (5, 6) of the twelve clinkers are given in Table I. Clinkers Nos. 16839, 16843, and 16890 are the products of one plant; clinkers Nos. 16827 and 16831 are the products of another plant; and the remaining 7 clinkers are each from a different plant.

Five of the clinkers were ground in commercial mills to obtain cements of widely different specific surface: approximately 1200, 1800, and 2300 sq. cm. per g. For these grinds gypsum was added to the clinker before grinding to give about 1.8 per cent SO_3. These cements were used to study the influence of fineness on the rate of hydration of cements with SO_3 constant.

For a study of variations in SO_3 contents, additional cements were ground in a laboratory mill from the twelve clinkers mentioned above. A natural rock gypsum was used which by chemical analysis was known to be almost pure $CaSO_4 2H_2O$. Three grinds were made with each clinker: (1) without gypsum, (2) with gypsum added to give an SO_3 content of 1.90 per cent, and (3) with gypsum added to give an SO_3 content of 5.00 per cent. The cements were then blended in the proper proportions to give the desired intermediate SO_3 contents. In these laboratory grinds the clinker with no added gypsum was ground to 1800 ± 25 sq. cm. per g. The specific surface of the cements containing gypsum were ground finer by 50 sq. cm. per g. for each 1.0 per cent of added SO_3, in an attempt to maintain the same fineness for the clinker in all grinds. This procedure was based on previous work by Swenson and Flint (7) who found that approximately 70 per cent of the gypsum in cement was present in the 0 to 7 μ fraction. Thus, if all were ground to the same specific surface, with increasing quantities of gypsum, the gypsum would contribute a disproportionate share of the total specific surface with the result that the specific surface of the clinker would become progressively lower as the SO_3 content increased.

THE INFLUENCE OF GYPSUM UPON THE RATE OF HEAT LIBERATION

Typical Results:

The rate of heat liberation was determined for the twelve commercial clinkers ground with various SO_3 contents as described above. The pastes were mixed and cured at 75 F. and the rate of heat liberation was determined for the first 72 hr. The temperatures of the cement and water were adjusted so that the temperature of the paste would be 75 F. when placed in the calorimeter. The room containing the calorimeters was maintained at 75 F.

A few typical results of the rates of reaction of the different cements are given in Figs. 4, 5, and 6, the rates of reaction being expressed in terms of the rate of heat liberation. The reactions of all cements undergo two cycles of increasing and decreasing rates, and cements of high or moderately high $3CaO\cdot Al_3O_3$ regardless of their alkali content or cements of low $3CaO\cdot Al_2O_3$ that are high in alkalies sometimes undergo a third cycle; whether the third cycle will occur depends on the SO_3

TABLE I.—CHEMICAL ANALYSES AND CALCULATED COMPOUND COMPOSITIONS OF CLINKERS.

Clinker	Oxide Analyses									Calculated Compound Composition						
	SiO_2	Al_2O_3	Fe_2O_3	Total CaO	MgO	SO_3	Ignition Loss	Na_2O	K_2O	C_3S	C_2S	C_3A	C_4AF	$CaSO_4$	Free CaO	MgO
CLINKERS OF HIGH $3CaO\cdot Al_2O_3$ AND LOW ALKALI CONTENT																
No. 15367	21.87	6.83	2.23	65.30	2.63	0.41	0.15	0.17	0.16	45.5	28.4	14.3	6.7	0.7	0.98	2.63
No. 16823	21.17	6.35	2.51	68.33	0.87	0.05	0.26	0.05	0.25	70.0	8.0	12.5	7.6	0.1	0.21	0.87
CLINKERS OF HIGH OR MODERATELY HIGH $3CaO\cdot Al_2O_3$ AND HIGH OR MODERATELY HIGH ALKALI CONTENT																
No. 15900	22.14	6.46	2.38	63.66	3.78	0.22	0.50	0.36	0.50	41.4	32.2	13.1	7.3	0.4	0.46	3.78
No. 15498	20.92	5.82	2.53	67.71	1.36	0.19	0.85	0.31	0.42	62.5	13.0	11.2	7.7	0.3	2.71	1.36
No. 16843	22.34	5.76	2.57	65.22	1.95	0.52	0.52	1.03	0.06	48.2	27.8	11.0	7.8	0.9	1.07	1.95
No. 15699	22.72	5.38	2.48	64.06	3.43	0.20	0.37	1.17	0.46	47.9	29.0	10.1	7.6	0.3	Nil	3.43
No. 16827	22.81	4.99	3.03	63.74	2.70	0.39	0.60	0.04	1.37	46.0	30.6	8.1	9.3	0.7	0.25	2.70
CLINKERS OF LOW $3CaO\cdot Al_2O_3$ AND LOW ALKALI CONTENT																
No. 16890	26.07	3.93	3.16	63.99	1.28	0.46	0.57	0.32	0.02	28.0	53.5	5.1	9.6	0.8	0.44	1.28
No. 15623	23.30	4.58	4.35	65.01	1.44	0.03	1.05	0.05	0.18	47.6	30.9	4.8	13.2	0.1	0.73	1.44
No. 15670	27.95	2.07	1.87	65.61	1.83	0.17	0.26	0.05	0.23	37.4	51.9	2.4	5.7	0.3	0.23	1.83
CLINKERS OF LOW $3CaO\cdot Al_2O_3$ AND HIGH ALKALI CONTENT																
No. 16839	24.02	5.28	4.60	62.13	1.56	0.67	0.53	1.01	0.10	26.0	49.1	6.2	14.0	1.1	0.12	1.56
No. 16831	21.28	5.27	4.84	61.87	3.12	0.53	1.29	0.05	1.37	46.0	26.3	5.7	14.7	0.9	0.07	3.12

[a] Corrected for free CaO, but not corrected for minor constituents.

NOTE: Clinker No. 15699 was made from a special batch of slurry prepared to produce a clinker of unusually high Na_2O content. The clinkers normally produced at the plant do not have this high Na_2O content.

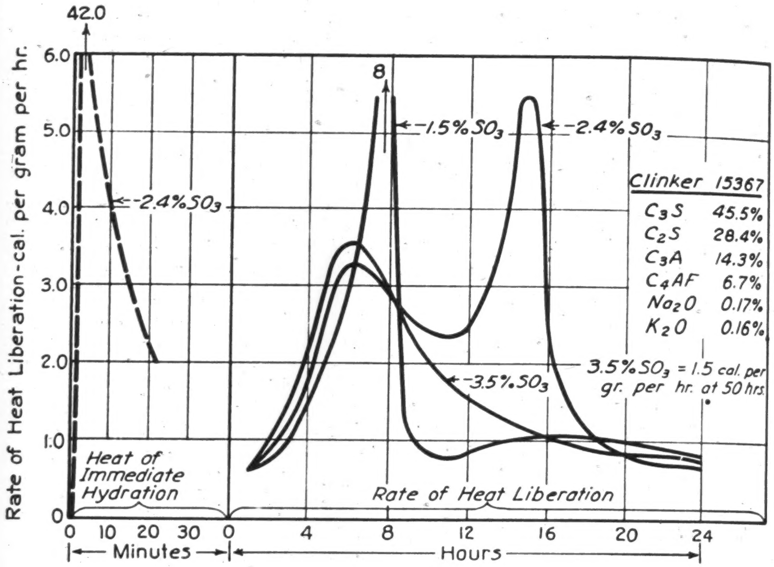


FIG. 4.—Rate of Hydration of Cements of High $3CaO \cdot Al_2O_3$ and Low Alkali Content With SO_3 Varied.

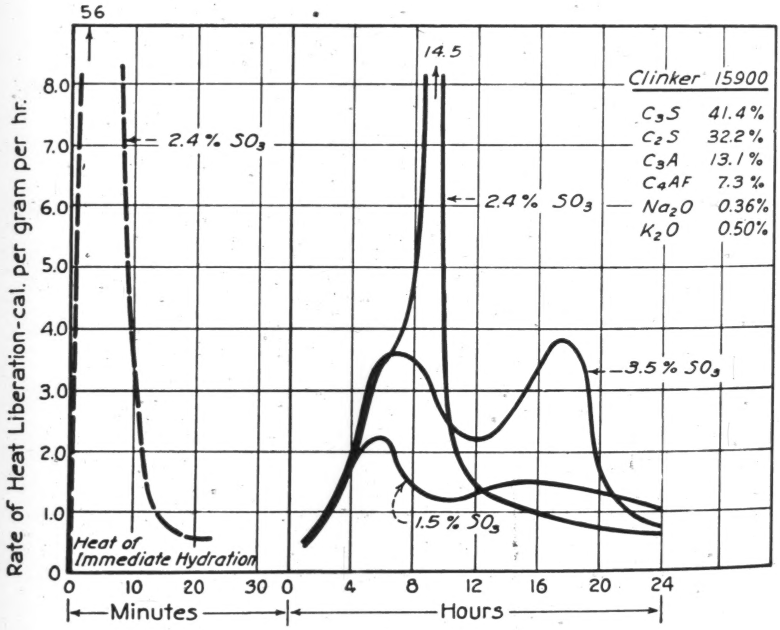


FIG. 5.—Rate of Hydration of Cements of High $3CaO \cdot Al_2O_3$ and Moderately High Alkali Content With SO_3 Varied.

content of the cement. During the first cycle shown by the broken line curves, the rate of heat liberation increases from zero to a relatively high value within the first five minutes and then decreases rapidly. This first cycle is designated the heat of immediate hydration. With cements of high or moderately high $3CaO \cdot Al_2O_3$ regardless of their alkali content or cements of high alkalies regardless of their $3CaO \cdot Al_2O_3$ content when ground without added gypsum or with small additions of gypsum 1.0 per cent SO_3 or less, the reactions are so rapid at this stage that flash-set occurs during mixing. With larger additions of gypsum the heat-liberation is decreased the reactions are slower) and the paste remains plastic. With cements of low $3CaO \cdot Al_2O_3$ and low alkali content even when ground without added gypsum the reactions are not sufficiently rapid to cause flash-set; however, gypsum will decrease their heat-liberation at this stage.

When the cements have sufficient gypsum to prevent the flash-set, the first cycle of rapid reaction is followed by a period of one or two hours of relatively slow reaction. During this period the paste retains much of its original plasticity. Following the interval of slow hydration, the second cycle of increasing rates of hydration occurs as shown by the first ascending portion of the solid line curves in Figs. 4, 5, and 6. This is the stage of initial hardening. With the cement of low $3CaO \cdot Al_2O_3$ and low alkali without added gypsum, Fig. 6, the period of slow hydration is abnormally long and it is shortened by the addition of gypsum. The third cycle of increasing rates of reaction is shown by the second ascending portion of the solid line curve for the cement containing 2.4 per cent SO_3 in Fig. 4 and the cement with 3.5 per cent SO_3 in Fig. 5. The behavior during the second and third cycles of increasing rates of reaction may differ widely among different cements. The significant characteristics of a cement to develop its highest strength and lowest contraction are revealed by the characteristics of the heat-liberation curves during this period.

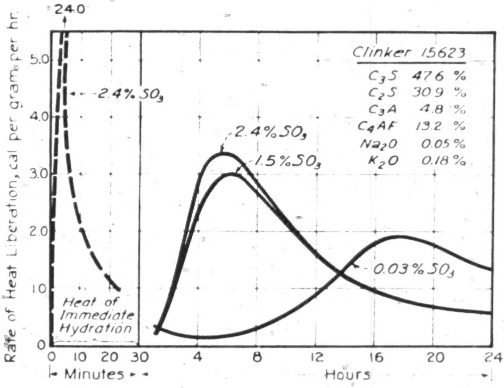


FIG. 6.—Rate of Hydration of Cements of Low $3CaO \cdot Al_2O_3$ and Low Alkali Content With SO_3 Varied.

The Rate of Hydration of a Properly Retarded Cement:

The curve for the cement of low $3CaO \cdot Al_2O_3$ and low alkali content with 1.5 per cent SO_3, Fig. 6, shows two cycles of increasing and decreasing rates of reaction and larger additions of gypsum do not appreciably alter the shape of the curve. Similar curves are obtained with clinkers of high $3CaO \cdot Al_2O_3$ content by the use of larger additions of gypsum. These curves are similar to the curves obtained for the hydration of $3CaO \cdot SiO_2$ either with or without gypsum. From these considerations and on the basis of the results of physical tests to be given later, a properly retarded cement can be considered as one which contains the minimum quantity of gypsum required to give a curve that shows two cycles of ascending and descending rates of heat liberation and that shows no appreciable change with larger additions of gypsum during the first 30 hr. of hydration. It will be shown later that cements containing the proper amount of gypsum to give this type of heat-liberation curve are the cements that have the highest strength and lowest contraction on drying. With cements of low $3CaO \cdot Al_2O_3$ content the shape of the curve after 30 hr. will not be altered by larger additions of gypsum. With cements of high $3CaO \cdot Al_2O_3$ content larger additions of gypsum may cause the appearance of a third cycle of increasing rates of reaction after 30 hr. However, it will be shown later that these larger additions of gypsum give lower strengths, higher contractions and abnormal expansions. It is for these reasons that a properly retarded cement is defined on the basis of the shape of the curve showing the rates of hydration during the first 30 hr. and the use of the minimum quantity of gypsum which will give that shape.

THE FUNCTION OF GYPSUM IN CONTROLLING THE EARLY REACTIONS OF HYDRATION

It has been shown that gypsum influences the rate of hydration of portland cement and that its effect upon the rate of hydration is not the same for all cements. In the following discussion it will be shown that gypsum influences the rate of hydration by its presence in solution and that a saturated lime-gypsum solution retards the hydration of cements of high $3CaO \cdot Al_2O_3$ content and accelerates the hydration of cements of low $3CaO \cdot Al_2O_3$ content.

The Influence of Gypsum Upon the Rate of Hydration of Cements of High $3CaO \cdot Al_2O_3$ and Low Alkali Content:

In an investigation of the reactions of water on the calcium aluminates, Wells (8) observed that in aqueous solutions containing only lime and alumina, the solubility of alumina decreased with increasing concentrations of calcium hydroxide. He observed also that in a saturated calcium hydroxide solution, alumina reacts with calcium hydroxide to form hydrated calcium aluminates. Lerch, Ashton, and Bogue (9) have shown that alumina is less soluble in a lime-gypsum solution than in lime-water and that the alumina reacts with lime and gypsum to form calcium sulfoaluminate.

With these facts on solubility in mind, the action of gypsum as a retarder can be explained on the basis of the solubility of alumina in the aqueous solutions with the aid of the curve obtained for the cement containing 2.4 per cent SO_3 in Fig. 4. When water is added to the cement there is an initial rapid solution of the anhydrous aluminates and a rapid crystallization of hydrated calcium aluminates. These are the rapid reactions which occur before the mixing water becomes saturated with lime and gypsum. Evidence of these reactions is given by the high rate of heat liberation observed during the first 5 min.—the rising portion of the broken-line curve. Simultaneously, the concentration of lime and gypsum increases and rapidly halts the reactions by decreasing the solubility of alumina—the descending portion of the broken-line curve. This prevents the immediate flash-set. The solution of lime alone is not enough to prevent flash-set, for experiments have shown that flash-set occurs in the absence of gypsum.

When the solution has become saturated with lime and gypsum, the anhydrous aluminate phases continue to dissolve slowly and react with gypsum to form calcium sulfoaluminate, and the anhydrous calcium silicates dissolve and form hydrated calcium silicates. These are the reactions of hydration in the retarded state. As these reactions occur, the rate of heat liberation again increases and reaches a maximum at about the sixth hour as shown by the solid-line curve. As the hydration proceeds the smaller anhydrous particles may become completely hydrated and the larger particles become smaller, that is, the surface area of anhydrous phases decreases. At the same time the hydration products are precipitated on the surface of the remaining anhydrous particles, partially sealing the grains. By a combination of these conditions, the rate of hydration decreases to the eleventh hour. As the gypsum continues to be used up in the formation of calcium sulfoaluminate, the gypsum eventually becomes depleted and its concentration in the solution decreases. When this condition is reached, the solubility of alumina again increases and a rapid reaction will occur if sufficient unhydrated calcium aluminates are available. This is shown by the third cycle of increasing rates of reaction from the eleventh to the fifteenth hour. The reactions that occur at this stage appear to be identical with those of the immediate flash-set that occurs in the absence of gypsum, but they are less rapid because of the decrease in surface area of

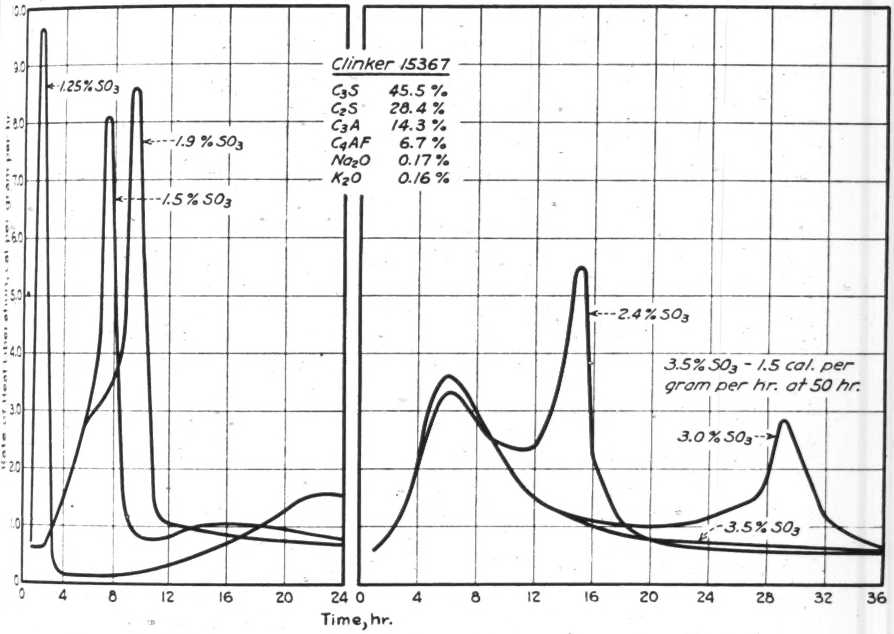


FIG. 7.—Rate of Hydration of Cements of High $3CaO \cdot Al_2O_3$ and Low Alkali Content With SO_3 Varied.

the anhydrous particles and because of the presence of the protective film of hydration products.

From what has just been said, it would be expected that the time at which the rapid hydration of the aluminate phases may occur will depend on the gypsum content of the cement. That this is actually the case is illustrated by the curves in Fig. 7 obtained with additional cements prepared from this same clinker of high $3CaO \cdot Al_2O_3$ content (clinker No. 15367). In Fig. 7 the peak appears at the second hour with 1.25 per cent SO_3, the tenth hour with 1.9 per cent SO_3, the fifteenth hour with 2.4 per cent SO_3, the twenty-ninth hour with 3.0 per cent SO_3 and at the fiftieth hour with 3.5 per cent SO_3. It is also shown that the peaks become lower with each progressive increase in SO_3 content.

TABLE II.—PARTIAL ANALYSIS OF EXTRACTS OBTAINED FROM HARDENED PASTES.
The cements used in the preparation of the pastes contained 3.0 per cent SO_3

Clinker	Partial Composition of Clinker, per cent			Partial Analysis of Extract after Period of Hydration Indicated, g. per 100 ml.					
				6 hr.			24 hr.		
	C_3A	Na_2O	K_2O	SO_3	Na_2O	K_2O	SO_3	Na_2O	K_2O
No. 15367	14.3	0.17	0.16	0.1207	0.0304	0.0582	0.0117	0.0381	0.0582
No. 15900	13.1	0.36	0.50	0.1576	0.0391	0.1457	trace	0.1952	0.2963
No. 15699	10.1	1.17	0.46	0.2142	0.1643	0.1351	0.0055	0.4333	0.2088
No. 15670	2.4	0.05	0.23	0.1120	0.0003	0.0216	0.1140	0.0116	0.0370
Saturated gypsum solution..........................				0.1220					

From these considerations it appears that the action of calcium sulfate as a retarder for cements of high $3CaO \cdot Al_2O_3$ content depends upon the decreased solubility of the anhydrous calcium aluminate in a saturated lime-gypsum solution. This observation was further confirmed by chemical analysis of the liquid phase extracted from hardened pastes and by expansion data obtained from mortar prisms.

Four cements, each containing gypsum equivalent to 3.0 per cent SO_3 were mixed in pastes with a water-cement ratio of 0.40 by weight. The pastes were placed in glass vials and allowed to harden. At ages of 6 and 24 hr. the pastes were pulverized and 200 g. of paste was mixed with 100 ml. of water. After a mixing period of 10 min., some of the clear water was filtered off and analyzed. Table II gives the partial analysis of the solutions. After 6 hr. of hydration, the concentration of dissolved SO_3 is approximately equal to that of saturated solution of gypsum, and with the cements of relatively high alkali content, Nos. 15900 and 15699, the concentration is even higher. The higher SO_3 concentrations can be accounted for by the presence of alkali sulfates formed by a reaction of the alkalies with gypsum. After 24 hr. of hydration, the concentration of dissolved SO_3 in the extracts from the three cements of high $3CaO \cdot Al_2O_3$ content is very low, and with the cement of low $3CaO \cdot Al_2O_3$ content, the concentration is very nearly that of a saturated gypsum solution. Thus, the results give further evidence of the rapid depletion of the gypsum in the pastes prepared from cements of high $3CaO \cdot Al_2O_3$ content, and the relatively slow depletion of the gypsum with cements of low $3CaO \cdot Al_2O_3$ content.

The reactions involved in the formation of calcium sulfoaluminate are known to be accompanied by expansion of the test specimen. Such expansion would be expected to continue as long as gypsum is available for the formation of calcium sulfoaluminate. With this in mind, mortar prisms containing cement 1.0 part, powdered silica 0.3 parts and standard Ottawa sand 2.3 parts by weight were made for length-change measurements. The pulverized silica was added to improve the plasticity of the otherwise harsh standard Ottawa sand mix. Cements of high $3CaO \cdot Al_2O_3$ content with SO_3 varied were used. The prisms were removed from the molds at 8 hr., and length-change measurements were made with the specimens cured in water at 70 F. The results are recorded in Table III which shows: (1) the time at which the gypsum was depleted as indicated by the heat-liberation curves, Fig. 7; (2) the expansion observed from 8 hr. to 24 hr., and from 24 hr. to 7 days. The length-change measurements do not register expansion that occurs before the initial measurement at 8 hr.

TABLE III.—EXPANSION OF MORTAR PRISMS CLINKER NO. 15367, SO_3 VARIED.

Mix: Cement 1.0, powdered silica 0.3, standard Ottawa sand 2.3 by weight.

SO_3 Content, per cent	Specific Surface, sq. cm. per g.	Hour When Gypsum Was Depleted (from heat curves)	Expansion, per cent	
			8 to 24 hr.	24 hr. to 7 days
1.5	1850	6th	0.005	0.009
1.9	1870	8th	0.006	0.009
2.4	1890	12th	0.012	0.007
3.0	1940	24th	0.032	0.005
3.5	2000	50th	0.043	0.026

These data show that: (1) when the heat curves indicate that the gypsum is depleted in 8 hr. or less (as with 1.5 or 1.9 per cent SO_3), the expansions from 8 hr. to 24 hr. and from 24 hr. to 7 days are low; (2) when the heat curves indicate that the gypsum is not depleted within 8 hr., the expansions between 8 and 24 hr. increase progressively with increasing SO_3 contents; (3) when the heat curves indicate that the gypsum is depleted within 24 hr., the expansions between 24 hr. and 7 days are low; (4) when the heat curves indicate that the gypsum is not depleted within 24 hr. (3.5 per cent SO_3), the expansions between 24 hr. and 7 days are greater than that obtained from similar compositions of lower SO_3 content. Thus the results obtained by the two methods of observation, rate of heat liberation and rate of expansion, are in excellent agreement as to the time at which the gypsum is depleted. The results also indicate the need of an upper limit on the gypsum content to avoid undue expansion. This will be discussed in more detail below.

Discussion of Previous Theories:

In an investigation of retarders reported in 1934 Roller (10) concluded that retardation results from the formation of a protective film of hydrated tetracalcium aluminate around the cement grains and that added gypsum reacts with the alkalies in the aqueous solution to form calcium hydroxide which promotes the formation of the tetracalcium aluminate. Forsen (11) concluded that retardation resulted from the formation of a protective film of tetracalcium aluminate or calcium double salts (calcium sulfoaluminates, etc.) He concluded also that in the absence of alkalies, lime alone was an adequate retarder, but with alkalies present gypsum was required to neutralize the alkalinity resulting from the solution of the alkalies. These investigators arrived at their conclusions from studies of the time of set as measured by the conventional penetration tests.

The theory of the formation of a protective film of hydration products does not adequately explain the data presented above on the rates of heat liberation. The hydration products may partially seal the particles and thereby partially be responsible for the reduction in rate of subsequent reactions. However, this phenomenon would not explain the occurrence of the subsequent rapid reactions that are coincident with the depletion of gypsum. For example, in Fig. 4 the curve for the cement with 2.4 per cent SO_3 shows a peak at 6 hr. followed by a decrease in the rate of reaction. If this decrease in the rate of heat liberation were to be explained entirely on the basis of the formation of a protective film of hydration products, it would be difficult to explain the subsequent disappearance of the protective film, as would be necessary, to permit the later rapid reaction and high rate of heat liberation. The peak in the curve at 15 hr. is actually higher than that at 6 hr.

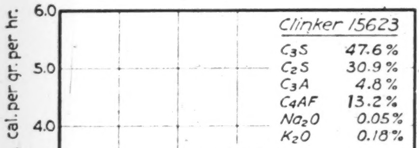


FIG. 8.—Rate of Hydration of Cements of Low $3CaO \cdot Al_2O_3$ and Low Alkali Content With SO_3 Varied.

However, these conditions can be explained from a consideration of the decreased solubility of alumina in a lime-gypsum solution.

The results obtained with another clinker of high $3CaO \cdot Al_2O_3$ and low alkali content, clinker No. 16823, are similar to those shown in Fig. 7. The clinker has a higher $3CaO \cdot SiO_2$ content than the one shown in Fig. 7. The higher $3CaO \cdot SiO_2$ appears to accelerate the over-all hydration with the result that the delayed rapid reaction occurs earlier and the peaks are somewhat higher for comparable SO_3 contents.

The Influence of Gypsum upon the Rate of Hydration of Cements of Low $3CaO \cdot Al_2O_3$ and Low Alkali Content:

The effect of SO_3 content upon the rate of hydration of cements prepared from a clinker of low $3CaO \cdot Al_2O_3$ content (4.8 per cent) and low alkali content, clinker No. 15623, is shown in Fig. 8. As stated previously there is no evidence of a flash-set in the absence of gypsum; and the addition of gypsum accelerates the rate of heat-liberation at early ages. To explain this acceleration, some additional facts must be called to mind. In a previous investigation (12) the author observed the accelerating effect of gypsum and explained the reactions on the basis of the formation of amorphous hydrated calcium ferrite and crystalline calcium sulfoferrite hydrate. In the absence of gypsum, $4CaO \cdot Al_2O_3 \cdot Fe_2O_3$ hydrates to form *crystalline* hydrated calcium aluminate and an *amorphous* hydrated calcium ferrite. It seems probable that the amorphous hydrated calcium ferrite, by precipitation on the cement particles, retards the subsequent hydration of the particles. It may be observed from the immediate heats of hydration, Fig. 15, the diagram at the right, that in the absence of gypsum the heat liberated within the first 5 min. is relatively high. It is apparently during this interval that rapid hydration of the $4CaO \cdot Al_2O_3 \cdot Fe_2O_3$ occurs and the hydrated calcium ferrite precipitates. MacIntire and Shaw (13) found that in the system $CaO—Fe_2O_3—SO_3—H_2O$ a crystalline calcium sulfoferrite is formed. It is probable that when gypsum is added to the clinker of low $3CaO \cdot Al_2O_3$ content, crystalline calcium sulfoferrite is formed in the cement paste and that in this state this compound does not seal the cement particles.

The results obtained with cements prepared from two additional clinkers of low $3CaO \cdot Al_2O_3$ and low alkali content (clinker Nos. 15670 and 16890) are very similar to those obtained from clinker No. 15623 in Fig. 8.

The Effect of the Alkalies on the Gypsum Requirements of Cements:

The effect of the alkali content of the cement upon the gypsum required for proper retardation cannot be definitely explained at this time. However, the experimental data will be presented and a tentative explanation will be given. The results obtained with cements of different $3CaO \cdot Al_2O_3$ content and varying alkali content indicate that the alkalies influence the rate of reaction of the cement. The characteristic differences brought about by the higher alkali content are: (1) That at some of the lower SO_3 contents the cement may give a heat-liberation curve somewhat similar to that of a properly retarded cement, whereas additional tests with higher SO_3 contents will show that they are not properly retarded; (2) That gypsum reacts more rapidly with the cements of higher alkali content; and (3) That higher SO_3 contents are required for proper retardation of such cements.

It seems probable that both the phase composition of the clinker and the concentration of alkalies in the aqueous solution influence the rate of hydration of the cement. The phase systems of portland cement clinker have not been completely solved and the state of combination of alkalies in the clinker is not entirely known. Insley and McMurdie (14) believe that at least part of the alkalies are combined as a lime-alumina-alkali compound with a small amount of $4CaO \cdot Al_2O_3 \cdot Fe_2O_3$ in solid solution. Some unpublished data obtained by the author have shown that the alkalies are readily soluble in the liquid phase of portland cement clinker and that a part of the alkalies are thus likely to be present in the glass. Kalousek, Jumper, and Tregoning (15) and Taylor (16) have found evidence that part of the K_2O in the clinker may be present as K_2SO_4. Taylor (17) reports that under conditions of crystalline equilibrium the compound $K_2O \cdot 23CaO \cdot 12SiO_2$ was found to exist in the region of the K_2O—CaO—Al_2O_3—SiO_2—Fe_2O_3 system in which portland cement compositions fall. Probably, however, not all of the K_2O is present as K_2SO_4 or $K_2O \cdot 23CaO \cdot 12SiO_2$ in commercial clinker since a state of crystalline equilibrium is seldom if ever fully attained.

That at least some of the alkali-containing phases are attacked rapidly by water is evidence by the relatively large amounts of alkalies in the aqueous solution within a few minutes after mixing. Roller (10) observed this phenomenon and Kalousek, Jumper and Tregoning (15) found that of the total alkalies present in the clinker from 1 to 53 per cent of the K_2O and from less than 1 to as much as 30 per cent of the Na_2O dissolved in the mixing water in 7 min. Roller observed also that in the hydration of clinker the solution of alkalies produced a high pH and decreased the $Ca(OH)_2$ concentration of the aqueous solutions; with the addition of gypsum double decomposition occurs by reaction of the gypsum with the alkali hydroxides and the pH of the solution is decreased.

The Effect of Alkalies on the Rate of Hydration of Cements of High $3CaO \cdot Al_2O_3$:

The results obtained with cements prepared from a clinker of moderately high $3CaO \cdot Al_2O_3$ and high alkali content are shown in Fig. 9. This cement is low in Na_2O and high in K_2O. The curve for the cements containing 1.0 to 1.5 per cent SO_3 show only moderate peaks at the fourth hour. As the SO_3 content is increased to 2.4 or 3.0 per cent the peaks on the curves become abnormally high and occur later. This phenomenon of increasing rates of hydration with increasing SO_3 content cannot be definitely explained at this time though it appears to be associated with the alkali content of the cement. It has been observed with several cements of high or moderately high $3CaO\cdot Al_2O_3$ and high alkali content but has not been observed with cements of high $3CaO\cdot Al_2O_3$ and low alkali content. The curves for the cements of the latter type with low SO_3 contents have high peaks as shown in Fig. 7. With larger quantities of gypsum the abnormally high peaks are eliminated with the cement of high alkali content and with 4.0 per cent SO_3 the curves meet the requirements of a properly retarded cement.

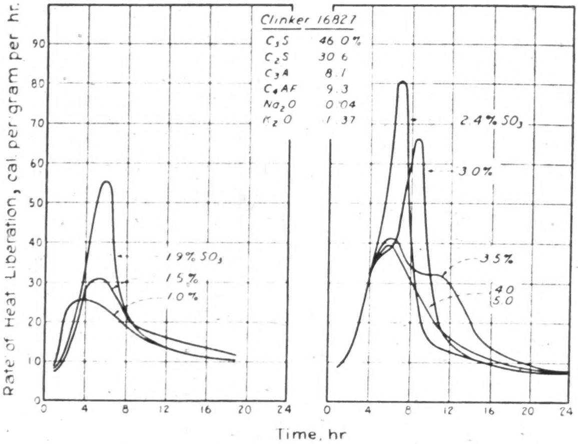


FIG. 9.—Rate of Hydration of Cements of Medium $3CaO\cdot Al_2O_3$ With Low Na_2O and High K_2O Content With SO_3 Varied.

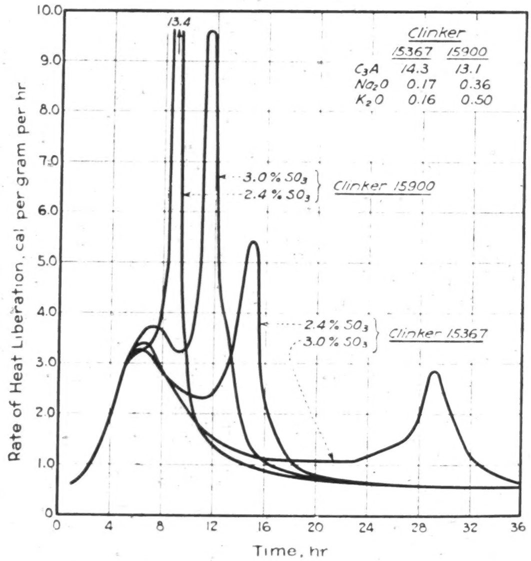


FIG. 10.—Effect of Alkali Content of the Cement in so far as it Influences the Effective SO_3 Content.

The results obtained with cements

prepared from four additional clinkers of high or moderately high $3CaO \cdot Al_2O_3$ and high alkali content (clinker Nos. 15900, 15699, 16843, and 15498) are similar to those described in Fig. 9.

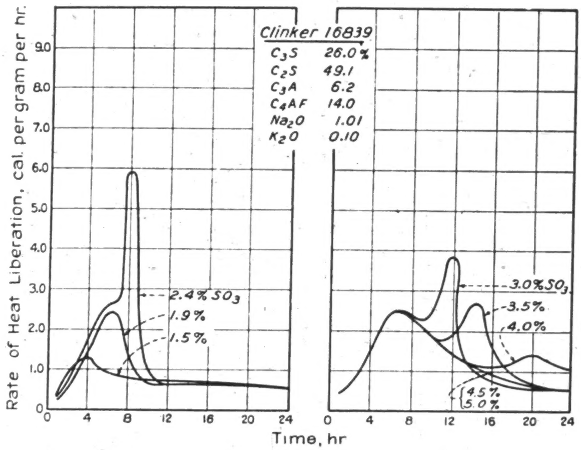


FIG. 11.—Rate of Hydration of Cements of Low $3CaO \cdot Al_2O_3$ With High Na_2O and Low K_2O Content With SO_3 Varied.

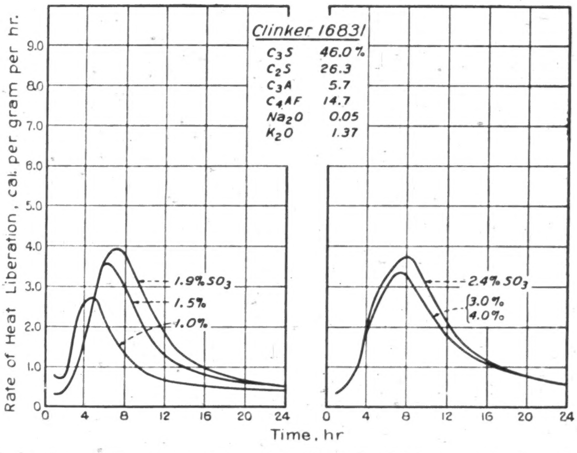


FIG. 12.—Rate of Hydration of Cements of Low $3CaO \cdot Al_2O_3$ With Low Na_2O and High K_2O Content With SO_3 Varied.

With cements of high $3CaO \cdot Al_2O_3$ content the gypsum reacts more rapidly with cements of high or moderately high alkali content than it does with cements of low alkali content. This is illustrated by Fig. 10 which compares the results obtained with cements of very similar composition except for a difference in alkali content. With the clinker of higher alkali content (No. 15900) the gypsum becomes depleted at the seventh hour for the cement containing 2.4 per cent SO_3 (indicated by the rapidly ascending portion of the curve at the seventh hour) and at the tenth hour with 3.0 per cent SO_3. On the other hand, with the clinker of low alkali content (No. 15367) the gypsum does not become depleted until the eleventh hour for the cement with 2.4 per cent SO_3 and the twenty-third hour with 3.0 per cent SO_3. With cements of high or moderately high $3CaO \cdot Al_2O_3$ content larger additions of gypsum are required for proper retardation as the alkali content of the cement increases. From what has just been said, it seems probable that at least part of the alkalies are present in the aluminate phases, and that aluminate phases containing alkalies react with gypsum more rapidly than do similar phases which are alkali free or of lower alkali content.

The Effect of Alkalies on the Rate of Hydration of Cements of Low $3CaO \cdot Al_2O_3$*:*

The results obtained with cements prepared from clinkers of low $3CaO \cdot Al_2O_3$ and high alkali content (Nos. 16839 and 16831) are shown in Figs. 11 and 12. The cements shown in Fig. 11 are high in Na_2O and low in K_2O, those in Fig. 12 are low in Na_2O and high in K_2O. These cements require large additions of gypsum for proper retardation and the cement high in Na_2O requires more gypsum than the cement high in K_2O. As the SO_3 content is increased from 1.0 or 1.5 per cent to 2.4 per cent the peaks on the curves become higher and occur somewhat later. The cements of high Na_2O content, Fig. 11 shows the third cycle of ascending and descending rates of hydration (the first cycle for the heat of immediate hydration is not shown) with 3.0, 3.5, and 4.0 per cent SO_3. The cements high in K_2O, Fig. 12, do not show the third cycle. With the clinkers of low $3CaO \cdot Al_2O_3$ content, the one high in Na_2O (Fig. 11) requires 4.5 per cent SO_3 to give the curve of a properly retarded cement, the one high in K_2O (Fig. 12) requires 3.0 per cent SO_3, while that of low alkali content (Fig. 8) require only 1.9 per cent SO_3.

The Effect of Alkalies on the Rate of Reaction of Gypsum with the Calcium Aluminates:

In the earlier discussion the action of gypsum in retarding the hydration of the aluminate phases was attributed to the fact that alumina is less soluble in a lime-gypsum solution than in a saturated lime solution. In the presence of the saturated lime-gypsum solution the aluminate phases dissolve at a retarded rate and react with lime and gypsum to form an insoluble calcium sulfoaluminate. By this process the gypsum eventually becomes depleted and its concentration in the aqueous solution decreases. When this condition is reached, the solubility of alumina again increases and a rapid reaction will occur if sufficient unhydrated calcium aluminates are available. This rapid reaction causes the abnormal peaks on the heat-liberation curves, sometimes observed as an abnormally high peak during the second cycle of ascending and descending rates and sometimes as a third cycle of ascending and descending rates. At this stage hydrated calcium aluminates are being formed in the paste. To bring out more clearly the relationship between the SO_3 concentration of the aqueous solutions and the appearance of the abnormal peaks on the curves some additional analyses were made of the aqueous solutions extracted from hydrating pastes. For this purpose two cements prepared from clinker No. 16839 were used, one with 3.5 per cent SO_3 and the other with 5.0 per cent SO_3. For these tests 200 g. of cement was mixed with 225 ml. of water and the paste was agitated continuously until the time of test. The clear liquid was then filtered off and analyzed. The partial analyses of the extracts are given in Table IV.

The results show that up to the seventh hour the compositions of the liquid extracts obtained from the two cements are almost identical, and during this time the rates of hydration of the two cements are identical, Fig. 11. At the twelfth hour the SO_3 concentration of the liquid extract obtained from the cement with 3.5 per cent SO_3 is considerably lower than it was at the seventh hour, and at the eighteenth hour it is practically nil. On the other hand, the SO_3 concentration of the liquid extract obtained from the cement with 5.0 per cent SO_3 increases from the seventh to the twelfth hour and from the twelfth to the eighteenth hour. The heat-liberation curves, Fig. 11 show that the rates of hydration of the two cements are almost identical up to the tenth hour. At the twelfth hour the third cycle of ascending rate of heat liberation is just starting for the cement with 3.5 per cent SO_3 and its rate of hydration then becomes higher than that of the cement with 5.0 per cent SO_3. Thus these results give further evidence that the gypsum functions as a retarder by its presence in the aqueous solution, and that the delayed rapid reaction occurs at the time the gypsum becomes depleted and its concentration in the aqueous solution decreases.

TABLE IV.—PARTIAL ANALYSES OF LIQUID EXTRACTS OBTAINED FROM HYDRATING PASTES.

Cements prepared from clinker No. 16839; C_3A, 6.2; Na_2O, 1.01; K_2O, 0.10 per cent.

Time of Extraction	Analysis, g. per liter			
	SO_3	CaO	Na_2O	K_2O
CEMENT WITH 3.5 PER CENT SO_3				
10 min.	2.181	1.325	1.974	0.402
4 hr.	2.137	1.370	2.214	0.425
7 hr.	2.404	1.235	2.567	0.450
12 hr.	1.444	0.485	3.259	0.518
18 hr.	0.002	0.130	4.949	0.560
CEMENT WITH 5.0 PER CENT SO_3				
10 min.	2.209	1.295	1.924	0.409
4 hr.	2.144	1.380	2.167	0.396
7 hr.	2.444	1.225	2.542	0.415
12 hr.	3.027	1.010	3.220	0.499
18 hr.	3.243	0.865	3.681	0.495

With cements of similar $3CaO \cdot Al_2O_3$ content the alkalies influence the rate of reaction of gypsum with the cements, those of high alkali content reacts with gypsum more rapidly than those low in alkalies. This observation is based on the results of analysis of liquid extracts given in Table V.

With the cement of high alkali content (No. 16839) the gypsum, equivalent to 3.5 per cent SO_3, has completely reacted in 18 hr. or less as shown by the absence of SO_3 in the extract, whereas with the cement of low alkali content (No. 16890) the gypsum has not completely reacted in 30 hr. These results seem to indicate that at least part of the alkalies are present in the aluminate phases, and that the aluminate phases containing alkalies hydrate more quickly than do aluminate phases which are alkali free or of lower alkali content. Thus, it seems probable that the effect of the alkalies on the gypsum requirements of cements of similar $3CaO \cdot Al_2O_3$ content may be ac-

TABLE V.—THE EFFECT OF THE ALKALIES ON THE RATE OF REACTION OF GYPSUM WITH CEMENTS OF SIMILAR $3CaO \cdot Al_2O_3$ CONTENT.

Time of Extraction, hr.	Partial Analyses of Liquid Extracts Obtained from Hydrating Pastes, g. per liter			
	SO_3	CaO	Na_2O	K_2O
CLINKER NO. 16839—C_3A, 6.2; Na_2O, 1.01; K_2O, 0.10; SO_3, 3.5 PER CENT				
4	2.14	1.37	2.21	0.43
7	2.40	1.24	2.57	0.45
12	1.44	0.49	3.26	0.52
18	0.00	0.13	4.95	0.56
30	0.00	0.12	5.19	0.55
CLINKER NO. 16890—C_3A, 5.1; Na_2O, 0.32; K_2O, 0.02; SO_3, 3.5 PER CENT				
4	1.19	2.08	0.35	0.06
7	1.20	1.90	0.49	0.06
12	1.26	1.70	0.64	0.10
18	1.31	1.57	0.77	0.09
30	0.93	1.22	0.86	0.11

counted for on the basis of their influence on the rate of hydration of the aluminate phases.

The Relative Effect of Gypsum and Plaster of Paris:

Forsen (11) concluded that, used as retarders, gypsum ($CaSO_4 \cdot 2H_2O$) and plaster of Paris ($CaSO_4 \cdot \frac{1}{2}H_2O$) would not behave in the same manner. He believed that with the addition of gypsum a saturated calcium sulfate solution was formed and under these conditions $3CaO \cdot Al_2O_3 \cdot CaSO_4 \cdot 12H_2O$ precipitated as a protective film on the cement particles; but with the addition of plaster of Paris a supersaturated calcium sulfate solution was formed with the result that $3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 31\frac{1}{2}H_2O$ crystallized from the solution without forming a protective film. Thus Forsen concluded that gypsum retards the set but that plaster of Paris does not.

TABLE VI.—EFFECT OF DEHYDRATION OF GYPSUM UPON THE HEATS OF IMMEDIATE HYDRATION.

Cement	SO_3 Content, per cent	Specific Surface, sq.cm. per g.	Condition of Cement	Immediate Heat of Hydration, cal. per g.			
				5m.	10m.	15m.	30m.
CLINKER No. 15367—C_3S, 45.5; C_2S, 28.4; C_3A, 14.3; C_4AF, 6.7							
No. 15754...	2.4	1800	Original	3.9	4.3	4.5	4.8
No. 15754...	2.4	1800	Heated at 250 F. for 18 hr.	4.1	4.6	4.7	5.0

To examine Forsen's hypothesis of the effect of gypsum and plaster of Paris upon the rate of heat liberation, a cement was selected which had been ground in a laboratory mill at a low temperature, 75 F. Little, if any, dehydration of gypsum would be expected at such a low grinding temperature. A portion of the cement was heated to 250 F. for 18 hr. to dehydrate the gypsum. The rates of heat liberation were determined for the original cement and the heated cement; the results are given in Table VI and Fig. 13. These results show that the dehydration of the gypsum caused no appreciable change in the rates of heat-liberation. Thus it appears that the difference in the time of set, as observed by Forsen, resulted from the set of plaster of Paris *per se*, and not from any difference in the chemical reaction of the cement.

The Effect of Specific Surface upon the Rate of Hydration:

The five commercial clinkers ground at the plant to three degrees of fineness were used to determine the effect of fineness upon the rate of heat liberation. The pastes were mixed and cured at 75 F. and the rates of heat liberation were determined for the first 72 hr. It will be shown from the results of these tests that: (1) The maximum rate of heat liberation becomes higher as the specific surface increases. (2) With cements of high $3CaO \cdot Al_2O_3$ content the maximum rate of heat liberation becomes abnormally high at the higher specific surface when the SO_3 content is maintained constant. When the SO_3 content of the cements in

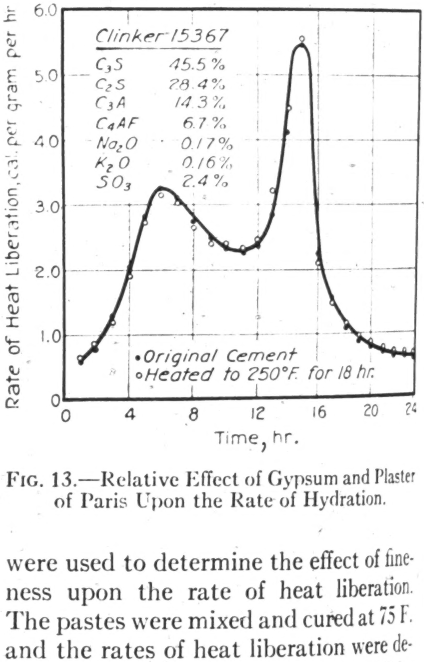


FIG. 13.—Relative Effect of Gypsum and Plaster of Paris Upon the Rate of Hydration.

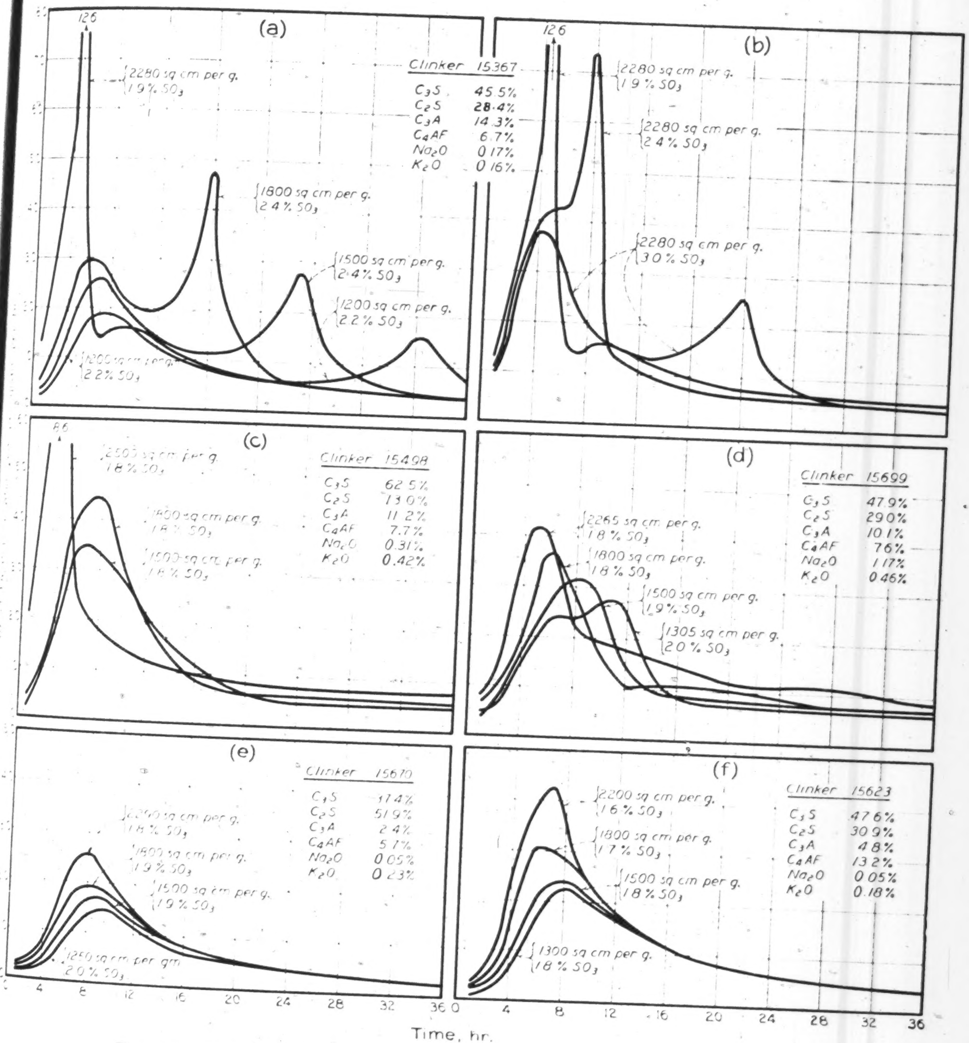


FIG. 14.—Effect of Specific Surface Upon the Heat of Hydration.

the higher range of specific surface is sufficiently high, the maximum rate of heat liberation is proportional to the increase in specific surface. (3) With cements of low $3CaO \cdot Al_2O_3$ content, the maximum rate of heat-liberation becomes higher by an amount proportional to the increase in the specific surface.

The results obtained with cements prepared from the clinkers of highest $3CaO \cdot Al_2O_3$ content (Nos. 15367 and 15498) are given in Fig. 14(*a*) and (*c*). With cements ground to specific surface of 1200, 1500, and 1800 sq. cm. per g., respectively, the rates of heat liberation within the first ten hours become progressively higher and become higher by amounts proportional to the increase in specific surface. With the cements prepared from clinker No. 15367 there is a third cycle of rapid heat liberation (the first cycle, the heat of immediate hydration is not shown in these figures) and as the specific surface is increased the peak of the third cycle becomes higher and occurs at an earlier age. With the cements of higher specific surface (2280 sq. cm. per g. in Fig. 14(*a*) and 2500 sq. cm. per g. in Fig. 14(*c*) the rate of heat liberation during the second cycle is abnormally high. The cement ground to 2280 sq. cm. per g. has a lower SO_3 content than the other cements shown in Fig. 14(*a*). Some additional tests were made with this cement in which powdered gypsum was added to the cement. The results are given in Fig. 14(*b*). When the SO_3 content is increased to 2.4 per cent the rate of reaction during the second cycle is decreased appreciably, but remains abnormally high as compared with the cement at 1800 sq. cm. per g. with the same SO_3 content. When the SO_3 content is increased to 3.0 per cent the rate of reaction during the second cycle is again decreased and is higher than the rate obtained at 1800 sq. cm. per g. by an amount approximately proportional to the increase in specific surface.

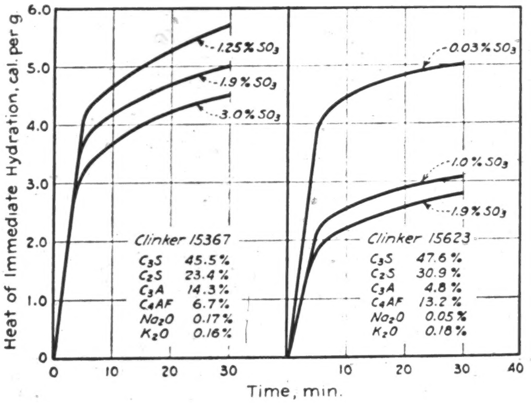


FIG. 15.—Heat of Immediate Hydration With SO_3 Varied.

The results obtained with cements prepared from the clinkers of lower $3CaO \cdot Al_2O_3$ content (Nos. 15699, 15623, and 15670) are shown in Fig. 14(*d*), (*e*), and (*f*). The effect of increasing the specific surface was unlike that when using cements high in $3CaO \cdot Al_2O_3$ content in that even with the highest specific surface no evidence of a deficiency of gypsum developed.

It is apparent that the results obtained from varying the specific surface are like those obtained by varying the SO_3 content. With cements of low 3CaO·

Al_2O_3 content, gypsum in amounts equivalent to 2.0 per cent SO_3 provides normal retardation for the range of specific surfaces investigated. Such increases in the rate of heat liberation as are observed are to be expected from the increased rate of hydration resulting from the higher specific surface. With cements of higher $3CaO \cdot Al_2O_3$ content it appears that with increasing specific surface, a larger portion of the aluminate phases becomes available for reaction at early ages and larger quantities of gypsum are required for proper retardation as measured by rates of heat-liberation.

The Heat of Immediate Hydration:

The curves showing heat of immediate hydration includes the heat liberated within 30 min. after water is added to the cement. The heats of immediate hydration were determined by means of the bottle calorimeter. The effect of SO_3 content upon the heats of immediate hydration for cements of the same specific surface is shown in Fig. 15. The heats of immediate hydration are higher for cements of high $3CaO \cdot Al_2O_3$ content than for cements of low $3CaO \cdot Al_2O_3$ content and become lower with increasing SO_3 contents. For cements of the same $3CaO \cdot Al_2O_3$ content variations in alkali content appear to have very little effect on the heats of immediate hydration. The effect of specific surface is shown in Fig. 16. The heats of immediate hydration become higher as the specific surface increases. A large percentage of the total heat liberated within the first 30 min. is liberated within the first 5 min. The results obtained for the heats of immediate hydration are in accord with the theory that the lime-gypsum solution is the effective retarder. With increasing quantities of gypsum the solution becomes saturated with gypsum more quickly, and the heat of immediate hydration is decreased. As the specific surface is increased with the gypsum constant, a larger portion of the aluminate phases is available for immediate reaction with the mixing water—before the solution becomes saturated with gypsum—and the heats of immediate hydration become larger.

Effect of Gypsum on the Cumulative Heats of Hydration:

Since gypsum alters the rate of hydration at early ages, it would be expected that the cumulative heats of hydration would be altered likewise. That this is true is shown by the date given in Table VII. The first additions of gypsum increase the heats of hydration while with larger additions of gypsum the values decrease. The quantity of gypsum required to obtain the highest heats of hydration is not the same for all clinkers, nor is it the same at all ages for the same clinker. The relationship between the heats of hydration at 1 and 3 days and the strengths at these ages will be discussed later.

THE INFLUENCE OF GYPSUM ON THE PHYSICAL PROPERTIES OF THE HARDENED PASTE

In the data already presented it has been shown that varying the gypsum content of the cement very definitely alters the rate of hydration at early ages and alters the hydration products that are formed. It seems probable that such changes in the hydration process would produce differences in the structure of the hardened paste and thereby alter the physical properties of the hardened paste. Previous investigators have found that gypsum influences the properties of portland cements. Kühl and Lu (18) found that shrinkage can be reduced by proper additions of gypsum and that the quantity of gypsum required to obtain the lowest shrinkage would increase with the $3CaO \cdot Al_2O_3$ content of the cement. Haegerman (19)

TABLE VII.—THE EFFECT OF GYPSUM ON THE CUMULATIVE HEATS OF HYDRATION—SERIES 287.
Neat cement pastes, water-cement ratio = 0.40 by weight.

SO_3, per cent	Heat of Hydration, cal. per g. for Period Indicated										
	5 min.	10 min.	30 min.	2 hr.	4 hr.	8 hr.	12 hr.	16 hr.	24 hr.	48 hr.	72 hr.
CLINKERS OF HIGH $3CaO\cdot Al_2O_3$ AND LOW ALKALI CONTENT Clinker No. 15367, C_3A, 14.3; Na_2O, 0.17; K_2O, 0.16 per cent											
1.5	4.1	4.6	5.3	6.6	9.0	24.8	28.2	32.4	39.8	54.3	63.5
1.9	3.8	4.2	5.0	6.3	9.3	20.9	35.8	39.7	46.2	59.2	68.1
2.4	3.5	3.9	4.6	6.1	9.5	21.5	31.1	45.9	53.0	65.5	73.8
3.0	3.3	3.8	4.5	5.9	9.2	20.9	28.8	33.7	42.3	65.1	72.8
3.5	3.3	3.7	4.5	6.1	10.0	23.1	30.9	35.6	42.2	59.8	75.1
Clinker No. 16823, C_3A, 12.5; Na_2O, 0.05; K_2O, 0.25 per cent											
1.0	3.6	4.1	5.3	6.9	10.5	17.7	19.4	22.6	33.7	58.6	72.1
1.5	3.3	3.9	4.8	6.4	10.5	32.4	37.4	42.8	51.4	67.0	76.1
1.9	3.1	3.7	4.6	6.1	10.6	37.5	44.3	49.1	56.3	68.8	77.1
2.4	3.1	3.6	4.4	5.9	10.3	30.0	51.0	55.5	61.9	72.8	79.5
3.0	3.1	3.5	4.4	5.8	10.1	28.8	42.4	51.8	60.4	71.7	79.5
3.5	3.0	3.5	4.4	5.8	9.8	28.2	40.4	47.5	57.3	71.5	78.8
4.0	3.1	3.5	4.3	5.7	9.6	27.5	39.7	45.8	53.4	69.5	77.6
CLINKERS OF HIGH OR MODERATELY HIGH $3CaO\cdot Al_2O_3$ AND HIGH OR MODERATELY HIGH ALKALI CONTENT Clinker No. 15900, C_3A, 13.1; Na_2O, 0.36; K_2O, 0.50 per cent											
1.5	4.3	4.8	5.4	6.7	9.4	16.8	23.4	27.0	37.0	53.6	66.1
1.9	4.7	5.4	5.9	7.1	9.7	27.0	33.2	38.1	45.5	60.6	70.3
2.4	4.7	5.0	5.8	7.1	10.2	24.3	44.2	48.4	54.4	68.4	74.7
3.0	4.2	4.6	5.3	6.6	9.8	23.5	45.1	53.4	59.3	69.7	75.6
3.5	4.0	4.5	5.1	6.6	10.0	23.9	34.0	45.2	60.2	70.6	76.6
Clinker No. 15498, C_3A, 11.2; Na_2O, 0.31; K_2O, 0.42 per cent											
1.0	3.7	4.1	4.8	8.0	14.3	21.8	28.1	32.1	42.0	60.1	72.8
1.5	3.5	3.9	4.6	7.1	13.9	29.3	35.5	39.2	45.2	59.3	70.0
1.9	3.2	3.5	4.3	6.8	13.2	29.2	39.1	43.6	49.4	63.1	73.7
2.4	3.2	3.6	4.3	6.9	14.2	31.1	39.7	45.5	53.8	69.5	78.4
3.0	2.9	3.2	4.1	6.9	15.0	32.4	40.4	45.5	52.9	66.2	76.1
Clinker No. 16843, C_3A, 11.0; Na_2O, 1.03; K_2O, 0.06 per cent											
1.5	4.0	4.6	5.7	8.0	12.2	17.8	22.6	28.1	37.1	55.2	67.2
1.9	4.0	4.4	5.3	7.1	12.0	24.6	30.4	35.0	42.1	59.2	68.7
2.4	3.7	4.3	5.2	7.4	13.0	33.0	41.1	45.6	52.1	67.8	73.3
3.0	3.7	4.1	5.1	7.6	13.9	28.4	40.3	48.7	59.3	69.0	72.9
3.5	3.7	4.1	4.9	7.0	12.9	27.7	35.8	42.1	52.7	67.7	72.0
4.0	3.6	4.0	4.8	7.0	13.3	28.0	35.2	39.8	47.0	65.3	72.1
4.5	3.3	3.8	4.6	6.7	14.3	29.9	37.0	40.9	46.1	53.8	57.9
Clinker No. 15699; C_3A, 10.1; Na_2O, 1.17; K_2O, 0.46 per cent											
1.0	2.6	3.5	4.6	6.7	10.1	19.5	27.1	32.6	40.6	59.9	68.9
1.5	2.6	3.4	4.2	5.7	9.9	22.8	28.6	33.3	41.8	61.4	68.9
1.9	2.8	3.4	4.1	6.2	10.8	26.8	33.1	38.2	46.5	65.2	71.5
2.4	2.7	3.3	4.0	5.8	10.1	31.4	39.5	45.0	53.6	70.5	74.7
3.0	2.8	3.3	4.0	5.8	10.1	27.6	42.6	48.1	56.3	69.0	73.0
Clinker No. 16827, C_3A, 8.1; Na_2O, 0.04; K_2O, 1.37 per cent											
1.0	3.3	5.1	6.8	9.8	14.9	23.8	29.7	34.7	42.6	57.8	65.4
1.5	3.6	4.4	5.2	7.2	11.6	22.2	28.4	34.2	42.8	58.3	65.9
1.9	3.4	4.1	5.3	7.2	12.9	28.3	34.2	39.4	47.2	61.9	69.3
2.4	3.3	4.0	4.8	6.9	12.2	34.6	41.1	45.5	52.0	64.9	70.6
3.0	3.3	3.9	4.7	6.6	11.4	28.7	43.2	47.4	53.3	63.4	67.8
3.5	3.2	3.8	4.7	6.9	12.1	27.5	40.2	47.5	55.0	65.1	69.7
4.0	3.3	3.8	4.7	6.6	11.4	25.5	33.6	39.2	47.4	61.9	69.1
CLINKERS OF LOW $3CaO\cdot Al_2O_3$ AND LOW ALKALI CONTENT Clinker No. 16890, C_3A, 5.1; Na_2O, 0.32; K_2O, 0.02 per cent											
1.0	2.6	3.0	3.7	4.9	6.5	8.9	12.1	15.3	20.8	29.7	34.8
1.5	2.4	2.8	3.4	5.1	9.3	18.8	22.7	24.9	28.4	34.9	39.0
1.9	2.4	2.9	3.5	5.2	9.8	18.9	24.8	28.4	32.4	37.9	41.2
2.4	2.6	2.9	3.5	5.1	9.6	18.7	23.9	27.2	31.8	38.9	41.8
3.0	2.3	2.7	3.4	5.4	10.3	19.3	24.1	27.1	31.0	37.7	42.5
3.5	2.4	2.8	3.5	5.3	9.6	18.8	23.9	26.8	31.4	36.3	39.4
4.0	2.3	2.7	3.4	4.7	9.1	17.9	22.6	25.3	28.5	33.5	37.0

TABLE VII.—*Continued*

SO₃, per cent	Heat of Hydration, cal. per g. for Period Indicated										
	5 min.	10 min.	30 min.	2 hr.	4 hr.	8 hr.	12 hr.	16 hr.	24 hr.	48 hr.	72 hr.
	Clinker No. 15623, C_3A, 4.8; Na_2O, 0.05; K_2O, 0.18 per cent										
0.03	4.0	4.5	5.0	5.6	5.9	6.7	9.1	15.0	28.1	44.6	51.9
1.0	2.3	2.5	3.1	4.0	7.7	18.5	24.4	27.8	32.8	39.4	44.1
1.5	2.0	2.3	2.9	3.9	7.8	19.2	27.1	31.6	37.0	45.2	49.6
1.9	1.9	2.2	2.8	3.8	7.9	20.3	28.4	33.6	39.8	48.5	53.4
2.4	2.0	2.3	2.8	4.1	8.1	20.5	28.2	32.9	38.9	48.2	53.7
	Clinker No. 15670, C_3A, 2.4; Na_2O, 0.05; K_2O, 0.23 per cent										
0.17	2.0	2.4	3.1	3.5	3.8	5.0	9.1	14.2	20.2	29.4	33.2
1.0	1.8	2.0	2.6	3.0	3.7	10.7	17.8	21.3	25.1	30.2	33.5
1.5	1.5	1.7	2.3	2.7	3.5	11.0	18.3	22.5	27.5	33.7	37.3
1.9	1.4	1.5	2.1	2.5	3.5	11.9	19.4	23.6	28.6	35.3	38.9
2.4	1.4	1.6	2.2	2.6	3.5	11.6	19.3	23.6	28.2	34.2	36.9
	CLINKERS OF LOW $3CaO \cdot Al_2O_3$ AND HIGH ALKALI CONTENT Clinker No. 16839, C_3A, 6.2; Na_2O, 1.01; K_2O, 0.10 per cent										
1.5	3.9	4.7	5.5	6.8	9.2	12.6	15.4	18.2	23.3	34.2	42.5
1.9	3.9	4.5	5.0	5.8	8.2	16.2	18.8	21.2	26.4	36.6	43.9
2.4	3.9	4.4	5.2	6.2	8.8	21.8	26.5	29.3	33.8	45.1	49.9
3.0	3.7	4.2	5.0	6.2	8.7	18.1	29.8	35.0	39.7	48.5	52.2
3.5	3.7	4.2	5.0	6.0	8.8	18.4	26.0	35.2	41.0	47.1	49.5
4.0	3.7	4.1	4.8	6.3	9.1	18.9	25.9	30.5	40.2	49.0	53.8
4.5	3.6	4.0	4.8	6.1	9.4	19.6	26.4	30.5	37.2	45.0	50.2
	Clinker No. 16831, C_3A, 5.7; Na_2O, 0.05; K_2O, 1.37 per cent										
1.0	1.5	2.3	3.7	5.2	9.6	17.8	21.2	23.7	27.3	37.6	44.2
1.5	2.0	2.6	3.7	4.4	6.9	19.5	26.9	30.5	35.1	43.6	48.5
1.9	2.0	2.7	3.7	4.5	7.1	21.0	31.0	36.0	40.9	49.6	54.3
2.4	2.0	2.6	3.7	4.7	7.7	21.0	32.3	38.0	43.8	53.2	58.3
3.0	2.0	2.7	3.6	4.3	7.5	20.3	29.9	35.9	42.8	53.3	58.5
3.5	2.0	2.7	3.5	4.5	7.1	18.5	27.0	32.1	38.5	48.9	54.9
4.0	1.5	2.2	3.4	4.4	7.3	19.2	27.9	32.5	38.1	47.5	52.6

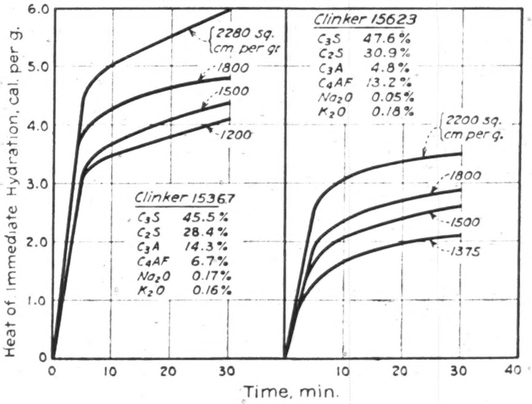


FIG. 16.—Heat of Immediate Hydration With Specific Surface Varied.

found that the quantity of gypsum required to obtain the highest strength and lowest shrinkage would depend on the $3CaO \cdot Al_2O_3$ content of the cement. With cements of high $3CaO \cdot Al_2O_3$ content the quantity of gypsum required would be larger than that permitted by present specifications.

No attempt was made in the tests reported here to examine the structure of

TABLE VIII.—STRENGTH AND MODULUS OF ELASTICITY OF MORTAR PRISMS—SERIES 287

One 2 by 2 by 9½-in. prism made on each of two days for each age of test.

Each flexural strength value and Young's modulus is the average of two tests, each compressive strength value is the average of four tests—modified 2-in. cubes.

Mix: Cement 1.0, pulverized silica 0.3, standard Ottawa sand 2.3.

Water content to give approximately 1-in. slump using a 6-in. cone.

SO_3, per cent	Water, gal. per sack	Flexural Strength, psi.						Compressive Strength, psi.						Young's Modulus of Elasticity, E, psi. $\times 10^{-6}$					
		1 day	3 days	7 days	28 days	3 months	1 yr.	1 day	3 days	7 days	28 days	3 months	1 yr.	1 day	3 days	7 days	28 days	3 months	1 yr.
CLINKERS OF HIGH $3CaO \cdot Al_2O_3$ AND LOW ALKALI CONTENT Clinker No. 15367, C_3A, 14.3; Na_2O, 0.16; K_2O, 0.17 per cent																			
1.5	5.02	195	575	770	890	845	880	630	2700	4610	6 650	6 940	6 990	1.7	3.7	4.6	5.3	5.3	5.6
1.9	5.02	290	675	815	1000	980	960	1000	2970	4830	7 250	8 240	8 380	2.3	3.9	4.7	5.3	5.5	5.7
2.4	5.02	320	670	860	1025	970	980	1380	3440	5320	7 830	8 370	8 840	2.7	4.1	4.8	5.5	5.7	6.0
3.0	5.02	275	640	870	1030	1100	1045	1300	3530	5610	7 830	8 940	8 700	2.5	4.1	5.0	5.5	5.9	6.0
3.5	5.02	270	640	905	990	1010	1020	1180	3740	5580	7 680	8 370	8 530	2.4	4.3	5.1	5.6	5.8	5.9
Clinker No. 16823, C_3A, 12.5; Na_2O, 0.05; K_2O, 0.25 per cent																			
1.0	4.82	105	805	1095	1035	1075	840	560	4360	6810	8 440	8 640	8 310	1.6	5.0	5.7	6.1	6.4	6.2
1.5	4.82	380	865	1185	1000	1045	945	1750	5560	7620	9 140	9 580	9 350	3.3	5.2	6.0	6.3	6.5	6.4
1.9	4.82	530	905	1055	1050	1050	1010	2160	5840	7910	9 980	10 080	9 180	3.6	5.3	6.0	6.5	6.5	6.4
2.4	4.82	590	935	1300	1245	1160	1110	2870	6330	8630	10 280	10 180	10 000	4.0	5.4	6.2	6.5	6.6	6.7
3.0	4.82	665	935	1030	990	1105	1040	3290	6460	8780	9 790	10 140	10 030	4.4	5.5	6.2	6.4	6.7	6.5
3.5	4.82	585	950	1085	1140	1120	1120	3250	6400	7990	9 780	9 320	9 960	4.4	5.5	6.2	6.4	6.4	6.5
4.0	4.82	510	925	1050	1130	1100	1060	2720	6360	8230	9 350	9 540	9 980	3.7	5.5	6.0	6.3	6.4	6.6
CLINKERS OF HIGH OR MODERATELY HIGH $3CaO \cdot Al_2O_3$ AND HIGH OR MODERATELY HIGH ALKALI CONTENT Clinker No. 15900, C_3A, 13.1; Na_2O, 0.36; K_2O, 0.50 per cent																			
1.5	4.85	215	565	720	890	1025	1015	700	3020	4440	6 850	7 940	7 980	1.9	4.0	4.6	5.8	5.9	6.1
1.9	4.85	325	635	795	885	960	990	1140	3330	4880	6 790	8 320	8 290	2.7	4.3	4.9	5.5	5.9	6.1
2.4	4.85	420	725	795	975	1055	950	1600	3760	5070	7 090	8 010	8 620	3.1	4.3	4.9	5.4	5.9	6.2
3.0	4.85	430	795	855	1005	1125	1095	2030	4210	5570	7 510	8 440	8 960	3.3	4.6	5.0	5.7	6.1	6.2
3.5	4.85	450	790	870	1045	1065	1085	2140	4410	5710	7 480	8 420	9 190	3.6	4.7	5.2	5.7	6.1	6.2
5.0	4.86	330	515	795	985	1115	1155	1610	3290	5220	7 760	9 280	10 390	2.9	4.2	5.1	5.8	6.3	6.4
Clinker No. 15498, C_3A, 11.2; Na_2O, 0.31; K_2O, 0.42 per cent																			
1.0	4.90	215	720	1030	975	855	915	900	4150	6800	7 820	8 200	8 140	1.7	4.2	5.3	5.6	5.6	5.8
1.5	4.90	360	785	1000	980	915	940	1480	4420	6860	8 360	8 490	8 620	2.6	4.4	5.4	5.7	5.8	6.0
1.9	4.90	445	830	1015	1085	995	955	1980	4720	7190	8 070	8 790	9 030	3.3	4.7	5.6	5.8	6.0	6.2
2.4	4.90	455	845	1125	1060	1020	1055	2140	5230	7280	8 390	8 950	9 300	3.4	4.9	5.7	5.9	6.0	6.2
3.0	4.90	380	835	1015	1170	1170	1035	2040	5240	7290	8 660	9 250	9 290	3.2	4.9	5.7	6.0	6.1	6.2
Clinker No. 16843, C_3A, 11.0; Na_2O, 1.03; K_2O, 0.06 per cent																			
1.5	4.70	285	750	920	1065	1045	1110	1010	3720	5360	7 030	7 460	8 380	2.4	4.7	5.6	6.3	6.6	6.7
1.9	4.70	400	860	925	960	1005	1105	1540	4180	5500	7 270	7 820	8 430	3.2	4.8	5.8	6.4	6.5	6.7
2.4	4.70	570	900	940	1050	1025	1025	2100	4640	5820	7 190	7 780	8 700	3.7	5.3	6.0	6.4	6.5	6.7
3.0	4.70	575	875	965	985	1085	1165	2640	4740	5820	7 110	7 560	8 420	4.1	5.5	6.1	6.3	6.5	6.8
3.5	4.70	555	850	1035	1030	1125	1105	2640	4880	5930	7 070	7 830	8 630	4.2	5.5	6.0	6.3	6.6	6.7
4.0	4.70	435	815	965	1020	1110	1170	2260	4960	6300	7 430	8 180	8 980	3.8	5.5	6.2	6.2	6.5	6.7
4.5	4.70	405	740	940	1040	1135	1180	2060	4530	6090	7 870	8 650	9 500	3.6	5.4	5.9	6.2	6.5	6.7
Clinker No. 15699, C_3A, 10.1; Na_2O, 1.17; K_2O, 0.46 per cent																			
1.0	5.00	215	635	850	880	875	860	940	3350	5200	6 130	6 620	6 880	2.0	4.3	5.1	5.5	5.7	5.8
1.5	5.00	265	655	880	880	980	960	1030	3490	5240	6 420	6 800	7 060	2.2	4.2	5.0	5.5	5.7	5.9
1.9	5.00	295	705	870	935	980	935	1200	3530	5200	6 180	6 340	6 790	2.4	4.4	5.2	5.3	5.5	5.8
2.4	5.00	310	660	910	945	925	950	1400	3550	5020	5 990	6 220	6 430	2.7	4.1	5.0	5.3	5.5	5.7
3.0	5.00	350	750	885	920	980	990	1560	3570	4940	5 530	6 100	6 330	2.9	4.3	5.0	5.1	5.3	5.6
5.0	4.96	390	855	920	1110	1065	1080	1860	4720	6140	6 480	7 450	8 200	3.1	4.9	5.6	5.9	5.1	6.2
Clinker No. 16827, C_3A, 8.1; Na_2O, 0.04; K_2O, 1.37 per cent																			
1.0	5.26	270	640	830	870	935	910	1000	3280	5120	6 540	6 760	6 990	2.1	4.2	5.0	5.5	5.9	5.9
1.5	5.12	285	705	865	960	980	970	950	3540	5360	6 820	6 960	7 520	2.2	4.3	5.2	5.7	5.8	6.0
1.9	5.00	345	740	915	855	1030	975	1310	3860	6070	7 060	7 260	8 020	2.8	4.6	5.5	5.8	6.0	6.1
2.4	5.00	405	840	995	990	1070	950	1680	4150	5900	7 140	7 480	8 190	3.2	4.7	5.6	5.9	6.1	6.2
3.0	5.00	525	785	1010	1065	1105	1085	2090	4350	5910	7 160	7 800	7 840	3.7	5.0	5.5	5.9	6.1	6.3
3.5	4.86	525	810	1140	1160	1090	1070	2380	4790	6130	6 940	7 700	8 230	4.0	5.2	5.8	6.2	6.3	6.4
4.0	4.86	390	860	1005	1050	1110	1230	2080	4620	5960	7 160	7 710	8 220	3.7	5.4	5.8	6.1	6.2	6.4

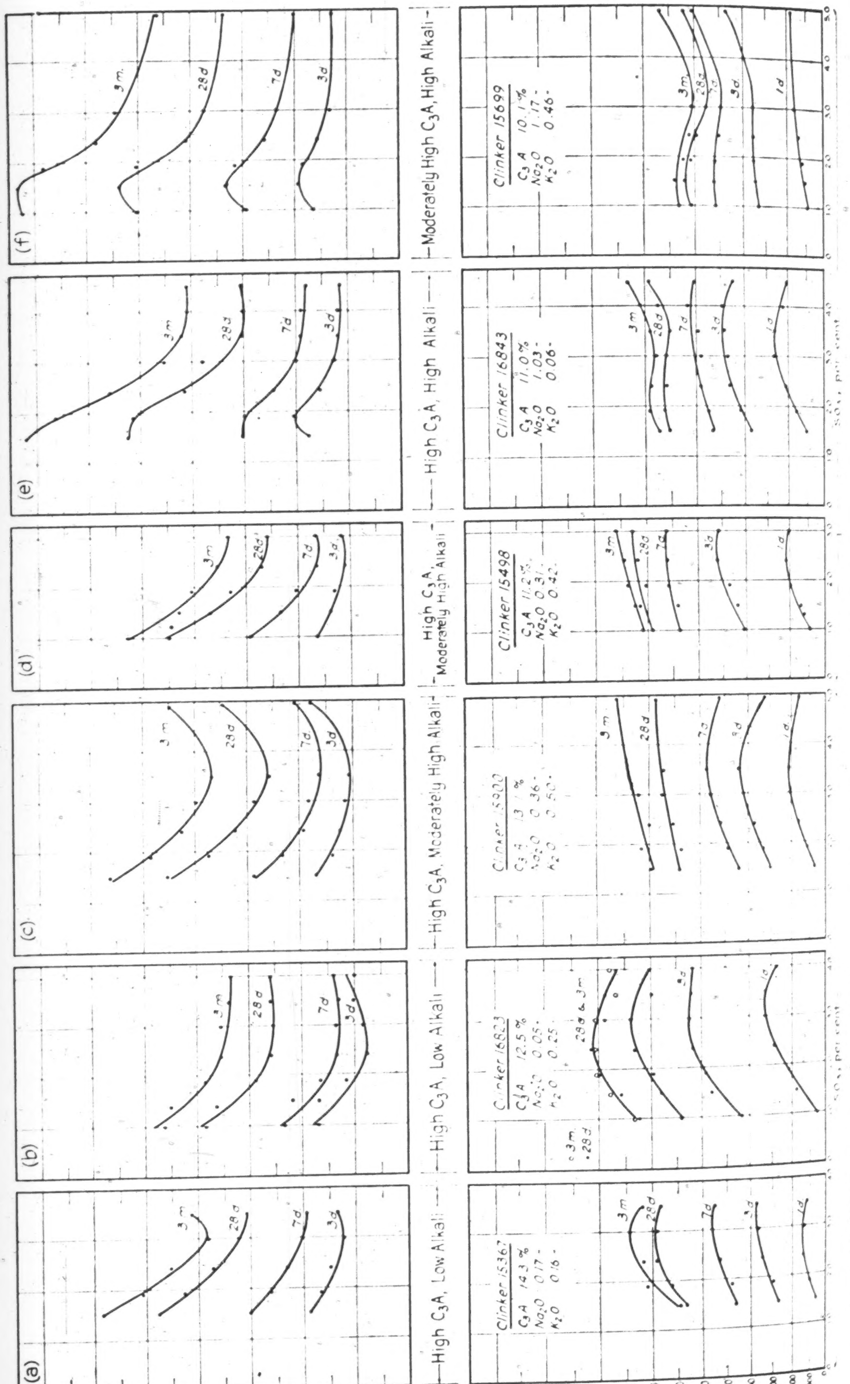
TABLE VIII.—*Continued*

SO_3, per cent	Water, gal. per sack	Flexural Strength, psi.						Compressive Strength, psi.						Young's Modulus of Elasticity, E, psi. 10^{-6}					
		1 day	3 days	7 days	28 days	3 months	1 yr.	1 day	3 days	7 days	28 days	3 months	1 yr.	1 day	3 days	7 days	28 days3	3 months	1 yr.
Clinkers of Low $3CaO \cdot Al_2O_3$ and Low Alkali Content Clinker No. 16890, C_3A, 5.1; Na_2O, 0.32; K_2O, 0.02 per cent																			
1.0	4.36	145	440	530	935	1010	1010	480	1570	2410	6 360	9 530	10 680	1.3	3.2	4.4	5.6	6.5	6.9
1.5	4.36	230	440	600	960	1190	1245	770	1700	2620	6 360	9 540	10 600	2.5	3.5	4.4	5.7	6.7	7.0
1.9	4.36	270	465	580	980	1225	1245	990	1860	2740	6 480	9 590	10 660	2.9	4.0	4.7	5.9	6.8	7.0
2.4	4.36	260	490	660	995	1220	1225	920	1970	2880	6 450	9 280	10 640	2.6	4.2	4.7	5.9	6.7	7.1
3.0	4.36	230	405	680	1025	1235	1240	820	1780	2990	6 280	8 860	10 320	2.3	3.8	4.8	6.0	6.7	7.1
3.5	4.36	200	380	610	995	1160	1160	820	1620	2840	6 130	9 160	10 290	2.3	3.6	4.7	6.0	6.8	7.0
4.0	4.50	215	375	560	920	1385	1125	780	1600	2540	6 190	9 100	10 370	2.1	3.4	4.5	5.9	6.5	7.0
Clinker No. 15623, C_3A, 4.8; Na_2O, 0.05; K_2O, 0.18 per cent																			
0.03	4.60	140	545	685	895	1005	1095	590	2820	4510	7 220	8 580	9 280	1.1	4.2	5.0	5.5	6.1	6.3
1.0	4.60	275	620	850	1055	1080	1165	1250	3180	4560	7 170	9 200	9 820	2.6	4.5	5.3	5.7	6.1	6.6
1.5	4.60	315	635	885	960	1195	1120	1410	3420	4910	7 060	9 140	10 020	2.8	4.5	5.2	5.7	6.2	6.5
1.9	4.60	315	625	810	970	1150	1255	1400	3460	4960	7 280	9 110	9 960	2.8	4.6	5.3	5.7	6.2	6.7
2.4	4.60	310	600	780	980	1115	1090	1480	3360	5100	7 360	9 270	10 370	2.7	4.4	5.2	5.7	6.3	6.5
Clinker No. 15670, C_3A, 2.4; Na_2O, 0.05; K_2O, 0.23 per cent																			
0.17	4.39	100	295	445	770	945	1090	390	1200	1980	4 710	8 020	8 560	1.5	2.8	3.7	5.2	6.2	6.3
1.0	4.39	215	390	545	945	1195	1190	780	1650	2390	6 120	10 150	10 320	2.1	3.4	4.2	5.5	6.0	6.8
1.5	4.39	210	410	585	895	1225	1235	840	1980	2780	6 170	10 250	10 670	2.3	3.8	4.4	5.7	6.6	6.8
1.9	4.39	190	385	580	1070	1205	1250	820	1880	2990	6 250	10 090	11 140	2.0	3.5	4.5	5.7	6.5	6.9
2.4	4.39	195	355	510	745	1060	1100	790	1500	2480	6 080	10 090	11 130	1.9	3.0	4.1	5.5	6.5	6.9
Clinkers of Low $3CaO \cdot Al_2O_3$ and High Alkali Content Clinker No. 16839, C_3A, 6.2; Na_2O, 1.01; K_2O, 0.10 per cent																			
1.5	4.70	65	360	490	835	1040	1040	360	1480	2400	5 230	6 940	8 040	1.1	3.0	3.9	5.4	6.1	6.4
1.9	4.61	40	405	585	940	1025	1005	460	1630	2640	5 250	7 500	8 520	1.4	3.2	4.1	5.6	6.3	6.7
2.4	4.70	145	490	610	910	995	1035	720	1840	2880	5 520	6 990	8 020	2.1	3.6	4.4	5.5	6.1	6.5
3.0	4.70	285	510	650	1110	1130	1065	950	2120	3130	5 500	7 140	8 520	2.6	3.9	4.6	5.7	6.3	6.6
3.5	4.70	345	535	715	1040	1030	970	1200	2240	3130	5 580	7 350	8 430	3.2	4.2	4.8	5.7	6.4	6.5
4.0	4.75	355	505	745	1135	1245	1150	1280	2270	3190	5 490	6 990	8 010	3.2	4.2	4.7	5.8	6.2	6.6
4.5	4.81	250	570	725	1040	1230	1305	1050	2290	3260	5 600	7 460	8 360	2.6	4.2	4.9	5.8	6.2	6.8
Clinker No. 16831, C_3A, 5.7; Na_2O, 0.05; K_2O, 1.37 per cent																			
1.0	4.61	230	485	750	1030	1015	1045	880	2320	4030	6 010	7 150	7 530	2.3	4.0	5.0	5.9	6.0	6.4
1.5	4.51	360	720	960	975	1000	1120	1550	3160	4800	6 480	7 280	7 800	3.3	5.0	5.7	6.2	6.3	6.6
1.9	4.51	490	775	905	1025	1015	1125	1920	3660	5080	6 470	7 660	8 100	3.8	5.1	5.8	6.2	6.4	6.7
2.4	4.51	510	790	995	1115	1125	1160	2100	4090	4960	6 580	7 180	7 820	4.1	5.3	5.8	6.2	6.4	6.6
3.0	4.56	455	825	925	1095	1075	1235	2220	4260	5400	7 000	7 830	8 560	4.0	5.4	5.8	6.2	6.6	6.7
3.5	4.56	425	745	915	1135	1070	1085	2030	4310	5680	6 970	7 880	8 880	3.7	5.3	5.9	6.2	6.6	6.6
4.0	4.56	375	655	730	985	1035	1150	1740	3470	4930	6 920	8 160	9 050	3.3	4.9	5.5	6.1	6.5	6.7

the hardened paste by direct methods. However, physical tests were made to determine the effect of variations in gypsum content upon the physical properties of the hardened paste. Mortar prisms were prepared for the physical tests. The properties determined for the prisms included: compressive strength, flexural strength, Young's modulus of elasticity by the electrodynamic method, expansion in water, and contraction in air. Miscellaneous tests of the cements included: specific surface, normal consistency, time of set, soundness and expansion in the autoclave test.

Preparation of Test Specimens:

The specimens used for the physical test were 2 by 2 by 9½-in. mortar prisms. Two specimens of a kind were used for each type of test. These were made in two rounds on different days. Those used for expansion in water and contraction in air were provided with gage points, one at each end of the long axis, for measurement of changes in length.


Contraction in Air, per cent
0.15
0.14
0.13
0.12
0.11
0.10
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
Compressive Strength, psi.
11,000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
(a)
(b)
(c)
(d)
(e)
(f)
High C3A, Low Alkali
High C3A, Low Alkali
High C3A, Moderately High Alkali
High C3A, Moderately High Alkali
High C3A, High Alkali
Moderately High C3A, High Alkali
Clinker 15367
C3A 14.3 %
Na2O 0.17 -
K2O 0.16 -
Clinker 16823
C3A 12.5 %
Na2O 0.05 -
K2O 0.25 -
Clinker 15900
C3A 13.1 %
Na2O 0.36 -
K2O 0.50 -
Clinker 15498
C3A 11.2 %
Na2O 0.31 -
K2O 0.42 -
Clinker 16843
C3A 11.0 %
Na2O 1.03 -
K2O 0.06 -
Clinker 15699
C3A 10.1 %
Na2O 1.17 -
K2O 0.46 -
3 m
28 d
7 d
3 d
1 d
28 d & 3 m
∘ 3 m
• 28 d
SO3, per cent

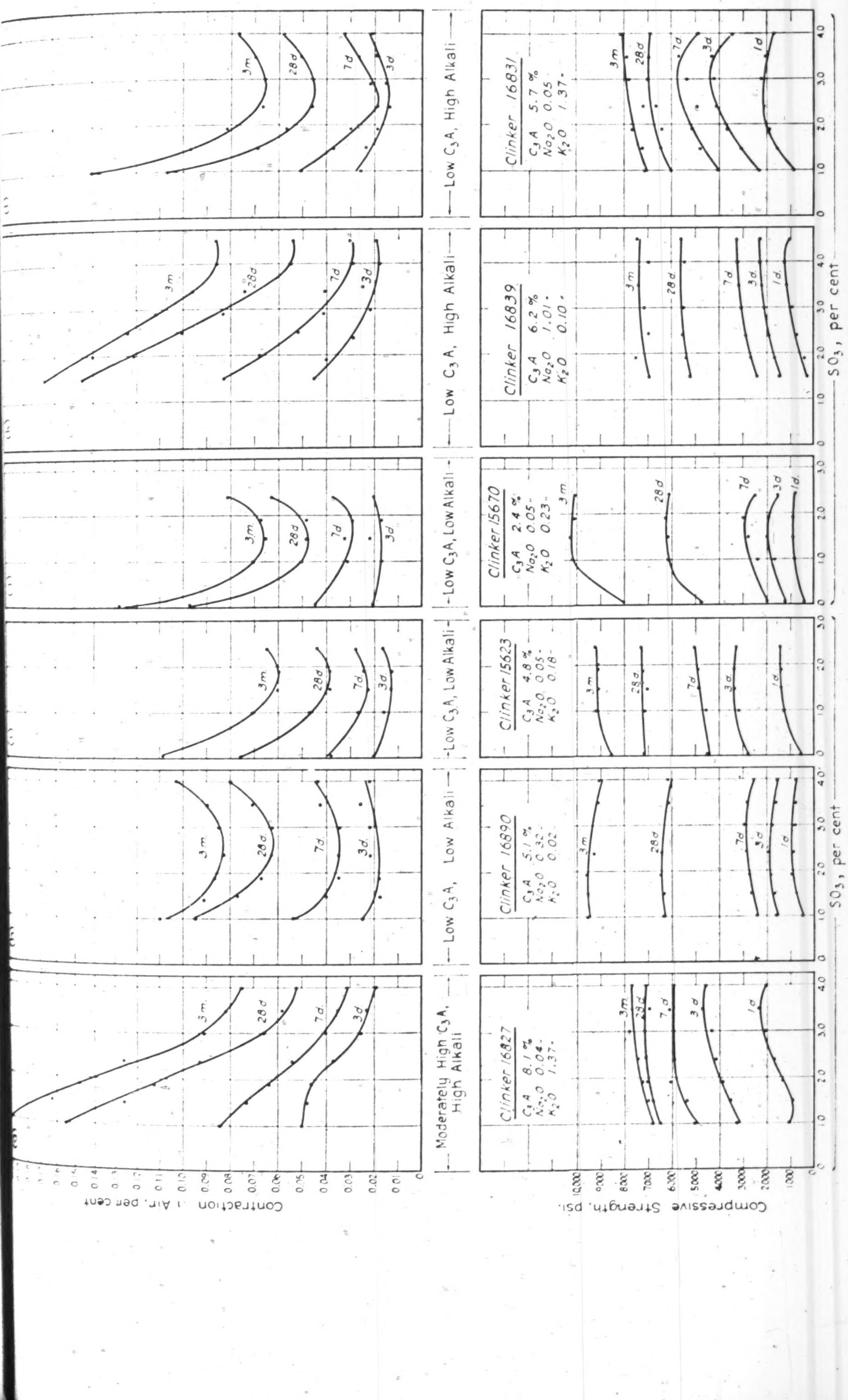


FIG. 17.—Influence of Gypsum on Contraction and Compressive Strength of 1:2.6 Mortars for Clinkers Nos. 15367, 16823, 15900, 15498, 16843, 15699, 16827, 16890, 15623, 15670, 16939, and 16831.

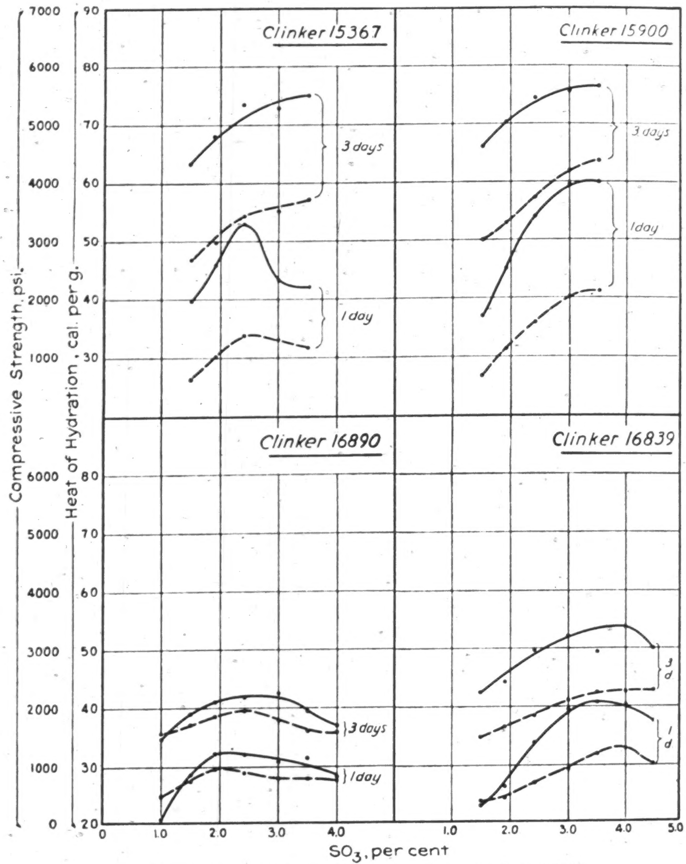


FIG. 18.—Relationship Between Heats of Hydration and Compressive Strengths at Early Ages.

TABLE IX.—THE INFLUENCE OF GYPSUM ON THE STRENGTH OF DIFFERENT $3CaO \cdot Al_2O_3$ AND ALKALI

SO$_3$, per cent	Compressive Strength of Modified Cubes, psi.											
	Clinker No. 15367			Clinker No. 15900			Clinker No. 15623			Clinker No. 16839		
	High $3CaO \cdot Al_2O_3$ Low Alkali			High $3CaO \cdot Al_2O_3$ Moderately High Alkali			Low $3CaO \cdot Al_2O_3$ Low Akali			Low $3CaO \cdot Al_2O_3$ High Alkali		
	1 day	7 days	1 yr.	1 day	7 days	1 yr.	1 day	7 days	1 yr.	1 day	7 days	1 yr.
0.03							590	4510	9 280			
1.0							1250	4560	9 820			
1.5	630	4610	6990	700	4440	7980	1410	4910	10 020	360	2400	6960
1.9	1000	4830	8380	1140	4880	8290	1400	4960	8 960	460	2460	7500
2.4	1380	5320	8840	1600	5070	8620	1480	5100	10 370	720	2880	6990
3.0	1300	5610	8700	2030	5570	9960				950	3130	7140
3.5	1180	5580	8530	2140	5710	9190				1200	3130	7350
4.0										1280	3190	6990
4.5										1050	3260	7460

The mix used was: cement 1.0 part, powdered silica 0.3 part, standard Ottawa sand 2.3 parts, by weight. The pulverized silica had a specific surface of 6000 sq. cm. per cu. cm. (Wagner method), which was very nearly the same as that of the cements.

The water content was adjusted with the intent of maintaining the slump (6-in. cone) approximately constant at 1 in. However, with only a limited quantity of cement available for trial mixes, the slumps obtained varied from 1 in. by as much as 0.5 in. For cements prepared from any one clinker, with the SO_3 content varied, the water content was maintained constant with only slight variations in the slump. However, for cements prepared from the different clinkers it was necessary to make rather large changes in water content to maintain a slump of approximately 1 in.

Each batch was mixed 1 min. dry, 2 min. wet, allowed to rest 3 min. and remixed 2 min. This schedule was adopted to eliminate the effect of premature stiffening, if any, on the water requirement The slump tests were made in duplicate, two operators working simultaneously on one batch from each round.

The specimens were cured in the molds in saturated air at 70 ± 2 F. for the first 2 hr.; the molds and contents were then immersed in water at the same temperature and allowed to remain there until the specimens were removed from the molds.

The Influence of Gypsum on Strength:

The strength tests at 1 day were made immediately after the prisms were removed from the molds. At the later ages they were made with prisms which were cured continuously in water at 70 ± 2 F. until the time of test. The flexural strength and Young's modulus of elasticity were determined on two prisms (one from each round) for each age, and the compressive strength was determined on the four half-prisms resulting from the flexural tests—tested as modified cubes. The results of the strength tests are given in Table VIII and Fig. 17.

The general characteristic of the influence of gypsum is that it increases the strength. However, the quantity of gypsum required to obtain the highest strength varies with the different clinker compositions. The results show that the alkalies as well as the $3CaO \cdot Al_2O_3$ influence the gypsum requirements of the cements. For cements of low alkali content those of high $3CaO \cdot Al_2O_3$ require larger additions of gypsum to give the highest strength than do those of low $3CaO \cdot Al_2O_3$ content. For cements of the same $3CaO \cdot Al_2O_3$ content those high in alkalies require larger additions of gypsum than those of low alkali content. Even cements of low $3CaO \cdot Al_2O_3$ content require relatively large amounts of gypsum when they are high in alkalies. These observations are illustrated by the compressive strength data in Table IX taken from the more complete data given in Table VIII.

With the two clinkers of low alkali content the best strengths are obtained for the one of high $3CaO \cdot Al_2O_3$ (No. 15367) with 2.4 to 3.0 per cent SO_3 and for the one of low $3CaO \cdot Al_2O_3$ (No. 15623) with 1.5 to 2.4 per cent SO_3. With clinkers of the high $3CaO \cdot Al_2O_3$ content the best strengths are obtained for the one low in alkalies (No. 15367) with 2.4 to 3.0 per cent SO_3 and for the one high in alkalies (No. 15900) with 3.5 per cent SO_3. With clinkers of low $3CaO \cdot Al_2O_3$ the best results are obtained for the one low in alkalies (No. 15623) with 1.5 to 2.4 per cent SO_3 and for the one high in alkalies (No. 16839) with 3.5 to 4.5 per cent SO_3.

Relationship Between the Heats of Hydration and Strength:

It has been shown that the first additions of gypsum increase the heats of hydration and strength at early ages whereas with larger additions of gypsum the values decrease. The relationship between the heats of hydration at 1 and 3 days and the compressive strengths at these ages for cements of different SO_3 contents is shown for four of the clinkers in Fig. 18. The results show a close relationship between the heats of hydration and strengths at these ages, that is, as the heats of hydration increase with increasing SO_3 contents the strengths increase, and as the heats of hydration decrease with the highest SO_3 contents the strengths decrease. Thus it appears that the effect of gypsum on the strengths at early ages can be accounted for, at least in part, by its effect on the hydration of the cements. However, results obtained at later ages show that a similar relationship no longer exists. The heats of hydration were determined at 28 days and 3 months for a few of the cements by the heat of solution method (20). The relationship between the heats of hydration and compressive strength at these ages is shown in Table X.

TABLE X.—HEATS OF HYDRATION AND STRENGTH OF CEMENTS OF DIFFERENT GYPSUM CONTENT.

SO_3, per cent	Heat of Hydration, cal. per g.				Compressive Strength, psi.			
	Clinker No. 15367		Clinker No. 15900		Clinker No. 15367		Clinker No. 15900	
	28 days	3 months	28 days	3 months	28 days	3 months	28 days	3 months
1.5	107.7	112.7	99.0	100.8	6650	6940	6850	7940
2.4	106.6	109.3	94.9	96.7	7380	8370	7090	8010
3.5	103.4	106.3	90.3	97.4	7680	8370	7480	8420

At these later ages the heats of hydration decrease while the strengths increase with increasing SO_3 contents. Thus it is evident that the effect of gypsum on strength cannot be accounted for entirely on the basis of its effect on the extent of hydration of the cements. It seems probable that the beneficial results obtained by the larger additions of gypsum are, at least in part, the result of its effect on the structure of the hardened paste.

The Influence of Gypsum on Expansion:

It appears that the purpose of limiting the SO_3 content of portland cement was to avoid a possible delayed expansion which may result from the reaction of gypsum with the calcium aluminates to form calcium sulfoaluminate. There does seem to be some basis for such a precaution as will be shown in data to be presented below. Furthermore in an investigation of the effect of finer grinding and higher SO_3 contents upon the physical properties of portland cement Bates (21) observed that for neat cement pastes increasing the SO_3 to 2.5 per cent increased the expansion of the coarse cements. However, in the same series of tests it was observed that with the finer cements there was very little increase in expansion with the higher SO_3 content up to the limit 2.5 per cent, the highest SO_3 content used in the series.

The results of the heat-liberation data and the extraction data cited previously have shown that, for cements of medium to high $3CaO \cdot Al_2O_3$ regardless of their alkali content, and cements high in alkalies regardless of their $3CaO \cdot Al_2O_3$ content, amounts of gypsum up to 3.5 per cent become depleted within 24 hr. when the cements are ground to a specific surface of 1800 sq. cm. per g. From these results it appears that the SO_3 content may be increased beyond the limit of present specifications without danger of delayed expansions from this source. The results of expansion measurements confirm this observation.

TABLE XI.—EXPANSION OF MORTAR PRISMS STORED IN WATER—SERIES 287.

One 2 by 2 by 9½-in. prism made on each of two days for each age of test.

Mix: Cement 1.0, pulverized silica 0.3, standard Ottawa sand 2.3.

Water content to give approximately 1-in. slump using a 6-in. cone.

SO_3, per cent	Water, gal. per sack	Expansion of Specimen Stored Continuously in Water for Period Indicated, per cent					
		7 days	14 days	28 days	3 mo.	1 yr.	2 yr.
CLINKERS OF HIGH $3CaO \cdot Al_2O_3$ AND LOW ALKALI CONTENT							
Clinker No. 15367, C_3A, 14.3; Na_2O, 0.16; K_2O, 0.17 per cent							
1.5	5.02	0.009	0.011	0.016	0.021	0.027	0.028
1.9	5.02	0.008	0.011	0.015	0.020	0.025	0.027
2.4	5.02	0.007	0.010	0.013	0.018	0.022	0.024
3.0	5.02	0.005	0.007	0.009	0.015	0.021	0.021
3.5	5.02	0.026	0.028	0.032	0.036	0.043	0.043
Clinker No. 16823, C_3A, 12.5; Na_2O, 0.05; K_2O, 0.25 per cent							
1.0	4.82	0.002	0.003	0.005	0.007	0.007	.....
1.5	4.82	0.001	0.000	0.003	0.006	0.006	.....
1.9	4.82	0.001	0.001	0.003	0.006	0.006	.....
2.4	4.82	0.001	0.004	0.005	0.009	0.008	.....
3.0	4.82	0.000	0.001	0.002	0.004	0.004	.....
3.5	4.82	0.001	0.004	0.004	0.006	0.007	.....
4.0	4.82	0.003	0.002	0.002	0.006	0.009	.....
CLINKERS OF HIGH OR MODERATELY HIGH $3CaO \cdot Al_2O_3$ AND HIGH OR MODERATELY HIGH ALKALI CONTENT							
Clinker No. 15900, C_3A, 13.1; Na_2O, 0.36; K_2O, 0.50 per cent							
1.5	4.85	0.007	0.009	0.013	0.023	0.039	0.039
1.9	4.85	0.005	0.009	0.015	0.021	0.038	0.038
2.4	4.85	0.006	0.010	0.012	0.018	0.034	0.034
3.0	4.85	0.005	0.007	0.011	0.017	0.033	0.032
3.5	4.85	0.002	0.005	0.009	0.015	0.033	0.030
5.0	4.86	0.053	0.053	0.060	0.061	0.071	0.073
Clinker No. 15498, C_3A, 11.2; Na_2O, 0.31; K_2O, 0.42 per cent							
1.0	4.90	0.020	0.020	0.027	0.032	0.043	0.042
1.5	4.90	0.017	0.017	0.025	0.031	0.038	0.035
1.9	4.90	0.015	0.016	0.023	0.028	0.038	0.035
2.4	4.90	0.013	0.013	0.020	0.026	0.037	0.034
3.0	4.90	0.022	0.022	0.026	0.033	0.042	0.039
Clinker No. 16843, C_3A, 11.0; Na_2O, 1.03; K_2O, 0.06 per cent							
1.5	4.70	0.004	0.016	0.022	0.027	0.034	.....
1.9	4.70	0.008	0.011	0.014	0.020	0.025	.....
2.4	4.70	0.010	0.011	0.012	0.017	0.022	.....
3.0	4.70	0.009	0.012	0.013	0.016	0.021	.....
3.5	4.70	0.003	0.004	0.004	0.006	0.011	.....
4.0	4.70	0.005	0.010	0.012	0.016	0.021	.....
4.5	4.75	0.011	0.017	0.018	0.021	0.027	.....
Clinker No. 15699, C_3A, 10.1; Na_2O, 1.17; K_2O, 0.46 per cent							
1.0	5.00	0.006	0.009	0.013	0.020	0.034	0.033
1.5	5.00	0.005	0.006	0.010	0.017	0.029	0.027
1.9	5.00	0.004	0.006	0.011	0.017	0.030	0.029
2.4	5.00	0.005	0.006	0.010	0.015	0.030	0.029
3.0	5.00	0.004	0.006	0.008	0.013	0.027	0.026
5.0	4.96	0.020	0.022	0.025	0.028	0.037	0.037
Clinker No. 16827; C_3A, 8.1; Na_2O, 0.04; K_2O, 1.37 per cent							
1.0	5.26	0.009	0.014	0.014	0.023	0.031	.....
1.5	5.12	0.010	0.010	0.012	0.019	0.027	.....
1.9	5.00	0.007	0.010	0.010	0.021	0.028	.....
2.4	5.00	0.007	0.009	0.011	0.020	0.026	.....
3.0	5.00	0.007	0.008	0.013	0.015	0.026	.....
3.5	4.86	0.008	0.008	0.011	0.016	0.019	.....
4.0	4.86	0.009	0.011	0.014	0.017	0.022	.....
CLINKERS OF LOW $3CaO \cdot Al_2O_3$ AND LOW ALKALI CONTENT							
Clinker No. 16890, C_3A, 5.1; Na_2O, 0.32; K_2O, 0.02 per cent							
1.0	4.36	0.010	0.012	0.016	0.021	0.025	.....
1.5	4.36	0.008	0.008	0.012	0.015	0.023	.....
1.9	4.36	0.014	0.010	0.012	0.018	0.024	.....
2.4	4.36	0.014	0.010	0.013	0.017	0.024	.....
3.0	4.36	0.025	0.020	0.022	0.028	0.032	.....
3.5	4.36	0.030	0.027	0.028	0.033	0.040	.....
4.0	4.50	0.023	0.027	0.029	0.037	0.045	.....
Clinker No. 15623, C_3A, 4.8; Na_2O, 0.05; K_2O, 0.18 per cent							
0.03	4.60	0.005	0.007	0.007	0.011	0.022	0.019
1.0	4.60	0.006	0.007	0.007	0.012	0.022	0.020
1.5	4.60	0.001	0.002	0.002	0.006	0.016	0.013
1.9	4.60	0.001	0.002	0.002	0.006	0.014	0.011
2.4	4.60	0.004	0.004	0.004	0.007	0.017	0.014
Clinker No. 15670, C_3A, 2.4; Na_2O, 0.05; K_2O, 0.23 per cent							
0.17	4.39	0.011	0.013	0.017	0.022	0.035	0.033
1.0	4.39	0.004	0.002	0.007	0.013	0.025	0.023
1.5	4.39	0.002	0.004	0.007	0.012	0.024	0.022
1.9	4.39	0.006	0.007	0.009	0.015	0.028	0.025
2.4	4.39	0.005	0.009	0.010	0.016	0.029	0.027
CLINKERS OF LOW $3CaO \cdot Al_2O_3$ AND HIGH ALKALI CONTENT							
Clinker No. 16839, C_3A, 6.2; Na_2O, 1.01; K_2O, 0.10 per cent							
1.5	4.70	0.007	0.009	0.010	0.015	0.023	.....
1.9	4.61	0.011	0.013	0.014	0.021	0.026	.....
2.4	4.70	0.010	0.011	0.015	0.018	0.027	.....
3.0	4.70	0.004	0.008	0.011	0.015	0.021	.....
3.5	4.70	0.005	0.007	0.008	0.014	0.019	.....
4.0	4.75	0.005	0.005	0.009	0.013	0.018	.....
4.5	4.81	0.007	0.007	0.010	0.015	0.019	.....
Clinker No. 16831, C_3A, 5.71; Na_2O, 0.05; K_2O, 1.37 per cent							
1.0	4.61	0.011	0.011	0.014	0.019	0.029	.....
1.5	4.51	0.008	0.011	0.014	0.017	0.025	.....
1.9	4.51	0.007	0.009	0.012	0.016	0.025	.....
2.4	4.51	0.005	0.017	0.008	0.014	0.020	.....
3.0	4.56	0.007	0.010	0.010	0.016	0.020	.....
3.5	4.56	0.008	0.008	0.010	0.014	0.022	.....
4.0	4.56	0.012	0.015	0.016	0.021	0.027	.....

The results of the length changes of the mortar prisms cured continuously in water at 70 ± 2 F. for 2 yr. are given in Table XI. The initial measurement was made at one day and the results are

TABLE XII.—CONTRACTION OF MORTAR PRISMS STORED IN AIR—SERIES 287.

One 2 by 2 by 9½-in. prism made on each of two days for each age of test.

Mix: Cement 1.0, pulverized silica 0.3, standard Ottawa sand 2.3.

Water content to give approximately 1-in. slump using a 6-in. cone.

Clinkers of High $3CaO \cdot Al_2O_3$ and Low alkali Content

Clinker No. 15367, C_3A, 14.3; Na_2O, 0.16; K_2O, 0.17 per cent

SO_3, per cent	Water, gal. per sack	Contraction in Air Storage for Period Indicated after Preliminary Curing 1 Day in Molds, 6 Days in Water, per cent						
		1 day	3 days	7 days	14 days	28 days	3 months	1 yr.
1.5	5.02	0.022	0.038	0.061	0.088	0.096	0.117	0.125
1.9	5.02	0.018	0.034	0.053	0.075	0.086	0.102	0.107
2.4	5.02	0.018	0.030	0.047	0.066	0.075	0.091	0.100
3.0	5.02	0.014	0.025	0.041	0.057	0.065	0.077	0.089
3.5	5.02	0.015	0.027	0.039	0.054	0.062	0.083	0.094

Clinker No. 16823, C_3A, 12.0; Na_2O, 0.05; K_2O, 0.25 per cent

SO_3, per cent	Water, gal. per sack	1 day	3 days	7 days	14 days	28 days	3 months	1 yr.
1.0	4.82	0.017	0.034	0.047	0.054	0.077	0.093	0.100
1.5	4.82	0.018	0.034	0.044	0.053	0.073	0.090	0.094
1.9	4.82	0.012	0.023	0.033	0.036	0.058	0.077	0.081
2.4	4.82	0.008	0.015	0.027	0.032	0.052	0.071	0.078
3.0	4.82	0.009	0.017	0.027	0.032	0.051	0.071	0.080
3.5	4.82	0.013	0.020	0.026	0.036	0.052	0.068	0.080
4.0	4.82	0.015	0.020	0.028	0.038	0.052	0.067	0.081

Clinkers of High or Moderately High $3CaO \cdot Al_2O_3$ and High or Moderately High Alkali Content

Clinker No. 15900, C_3A, 13.1; Na_2O, 0.36; K_2O, 0.50 per cent

SO_3, per cent	Water, gal. per sack	1 day	3 days	7 days	14 days	28 days	3 months	1 yr.
1.5	4.85	0.017	0.034	0.058	0.080	0.091	0.113	0.115
1.9	4.85	0.015	0.028	0.047	0.064	0.075	0.097	0.098
2.4	4.85	0.013	0.025	0.039	0.055	0.065	0.086	0.088
3.0	4.85	0.013	0.023	0.037	0.050	0.058	0.080	0.080
3.5	4.85	0.013	0.022	0.033	0.043	0.052	0.074	0.078
5.0	4.86	0.021	0.036	0.042	0.061	0.070	0.090	0.097

Clinker No. 15498, C_3A, 11.2, Na_2O, 0.31; K_2O, 0.42 per cent

SO_3, per cent	Water, gal. per sack	1 day	3 days	7 days	14 days	28 days	3 months	1 yr.
1.0	4.90	0.016	0.033	0.059	0.082	0.090	0.106	0.105
1.5	4.90	0.017	0.028	0.047	0.065	0.074	0.086	0.086
1.9	4.90	0.015	0.026	0.041	0.059	0.066	0.081	0.083
2.4	4.90	0.013	0.022	0.033	0.047	0.054	0.071	0.074
3.0	4.90	0.017	0.023	0.033	0.047	0.052	0.067	0.073

Clinker No. 16843, C_3A, 11.0; Na_2O, 1.03; K_2O, 0.06 per cent

SO_3, per cent	Water, gal. per sack	1 day	3 days	7 days	14 days	28 days	3 months	1 yr.
1.5	4.70	0.011	0.035	0.061	0.085	0.105	0.144	0.161
1.9	4.70	0.020	0.041	0.060	0.085	0.103	0.133	0.146
2.4	4.70	0.012	0.031	0.049	0.064	0.083	0.112	0.122
3.0	4.70	0.013	0.025	0.040	0.051	0.076	0.091	0.106
3.5	4.70	0.012	0.024	0.038	0.049	0.061	0.084	0.098
4.0	4.70	0.016	0.024	0.038	0.047	0.060	0.082	0.097
4.5	4.75	0.013	0.023	0.036	0.047	0.061	0.082	0.096

Clinker No. 15699, C_3A, 10.1; Na_2O, 1.17; K_2O, 0.46 per cent

SO_3, per cent	Water, gal. per sack	1 day	3 days	7 days	14 days	28 days	3 months	1 yr.
1.0	5.00	0.017	0.033	0.059	0.085	0.101	0.145	0.160
1.5	5.00	0.021	0.039	0.067	0.092	0.108	0.147	0.161
1.9	5.00	0.018	0.037	0.063	0.086	0.101	0.137	0.152
2.4	5.00	0.015	0.031	0.052	0.069	0.082	0.117	0.133
3.0	5.00	0.015	0.027	0.047	0.064	0.075	0.109	0.126
5.0	4.96	0.012	0.026	0.040	0.054	0.067	0.093	0.110

TABLE XII.—*Continued*

Clinker No. 16827, C_3A, 8.1; Na_2O, 0.04; K_2O, 1.37 per cent

SO_3, per cent	Water, gal. per sack	Contraction in Air Storage for Period Indicated after Preliminary Curing 1 Day in Molds, 6 Days in Water, per cent						
		1 day	3 days	7 days	14 days	28 days	3 months	1 yr.
1.0	5.26	0.025	0.050	0.085	0.118	0.155	0.201	0.230
1.5	5.12	0.023	0.048	0.073	0.099	0.126	0.163	0.177
1.9	5.00	0.017	0.046	0.064	0.089	0.113	0.148	0.165
2.4	5.00	0.023	0.037	0.054	0.074	0.093	0.126	0.143
3.0	5.00	0.014	0.026	0.041	0.055	0.066	0.101	0.119
3.5	4.86	0.014	0.023	0.035	0.045	0.058	0.082	0.105
4.0	4.86	0.012	0.019	0.031	0.040	0.052	0.075	0.097

Clinkers of Low $3CaO \cdot Al_2O_3$ and Low Alkali Content

Clinker No. 16890, C_3A, 5.1; Na_2O, 0.32; K_2O, 0.02 per cent

SO_3, per cent	Water, gal. per sack	1 day	3 days	7 days	14 days	28 days	3 months	1 yr.
1.0	4.36	0.008	0.025	0.054	0.079	0.095	0.110	0.110
1.5	4.36	0.008	0.018	0.040	0.062	0.077	0.091	0.100
1.9	4.36	0.006	0.018	0.035	0.053	0.067	0.087	0.093
2.4	4.36	0.009	0.022	0.035	0.048	0.063	0.083	0.094
3.0	4.36	0.013	0.022	0.035	0.047	0.063	0.085	0.097
3.5	4.36	0.009	0.026	0.043	0.057	0.071	0.090	0.107
4.0	4.50	0.010	0.022	0.044	0.062	0.080	0.103	0.114

Clinker, No. 15623, C_3A, 4.8; Na_2O, 0.05; K_2O, 0.18 per cent

SO_3, per cent	Water, gal. per sack	1 day	3 days	7 days	14 days	28 days	3 months	1 yr.
0.03	4.60	0.011	0.020	0.038	0.060	0.076	0.109	0.11
1.0	4.60	0.010	0.016	0.027	0.038	0.047	0.071	0.077
1.5	4.60	0.007	0.013	0.023	0.032	0.039	0.061	0.066
1.9	4.60	0.007	0.013	0.025	0.032	0.039	0.060	0.066
2.4	4.60	0.011	0.017	0.028	0.038	0.044	0.065	0.071

Clinker No. 15670, C_3A, 2.4; Na_2O, 0.05; K_2O, 0.23 per cent

SO_3, per cent	Water, gal. per sack	1 day	3 days	7 days	14 days	28 days	3 months	1 yr.
0.17	4.39	0.006	0.020	0.044	0.076	0.097	0.128	0.135
1.0	4.39	0.009	0.017	0.031	0.045	0.050	0.070	0.077
1.5	4.39	0.009	0.022	0.032	0.039	0.048	0.065	0.071
1.9	4.39	0.007	0.017	0.029	0.040	0.048	0.067	0.075
2.4	4.39	0.009	0.020	0.037	0.054	0.063	0.081	0.080

Clinkers of Low $3CaO \cdot Al_2O_3$ and High Alkali Content

Clinker No. 16839, C_3A, 6.2; Na_2O, 1.01; K_2O, 0.10 per cent

SO_3, per cent	Water, gal. per sack	1 day	3 days	7 days	14 days	28 days	3 months	1 yr.
1.5	4.70	0.011	0.045	0.083	0.117	0.145	0.166	0.171
1.9	4.61	0.013	0.040	0.068	0.099	0.121	0.140	0.150
2.4	4.70	0.007	0.029	0.052	0.081	0.101	0.124	0.140
3.0	4.70	0.006	0.022	0.041	0.058	0.083	0.112	0.124
3.5	4.70	0.011	0.025	0.041	0.054	0.074	0.106	0.120
4.0	4.75	0.008	0.018	0.029	0.040	0.055	0.086	0.103
4.5	4.81	0.009	0.019	0.030	0.042	0.054	0.086	0.104

Clinker No. 16831, C_3A, 5.7; Na_2O, 0.05; K_2O, 1.37 per cent

SO_3, per cent	Water, gal. per sack	1 day	3 days	7 days	14 days	28 days	3 months	1 yr.
1.0	4.61	0.010	0.026	0.051	0.077	0.107	0.141	0.161
1.5	4.51	0.014	0.024	0.037	0.055	0.069	0.097	0.115
1.9	4.51	0.009	0.019	0.030	0.045	0.057	0.082	0.100
2.4	4.51	0.005	0.014	0.019	0.032	0.046	0.067	0.082
3.0	4.56	0.007	0.016	0.022	0.032	0.046	0.066	0.080
3.5	4.56	0.008	0.019	0.027	0.037	0.051	0.070	0.084
4.0	4.56	0.008	0.021	0.033	0.044	0.058	0.077	0.091

recorded as the percentage expansion calculated from the length at 1 day. The results show that only a few specimens developed larger expansions as a result of the higher SO_3 content and that for these specimens the larger expansion occurs during the first few days. Clinker No. 15367 with 3.5 per cent SO_3, clinker No. 16890 with 3.0 to 4.0 per cent SO_3, and clinkers Nos. 15900 and 15699 with 5.0 per cent SO_3 show higher expansions at 7 days than do the corresponding cements of lower SO_3 contents prepared from the same clinkers. With these exceptions the expansions up to 1 yr. with as much as 3.5 to 4.5 per cent SO_3 are in no instance significantly higher than those with 1.9 per cent SO_3. Actually the least expansion is usually obtained with SO_3 contents greater than 1.9 per cent. Thus the results show that gypsum can be added to portland cements in considerably larger amounts than is permitted by current specifications without danger of abnormal expansion.

TABLE XIII.—THE INFLUENCE OF GYPSUM ON THE CONTRACTION OF MORTAR PRISMS.

SO_3, per cent	Contraction During Storage in Air for Period Indicated, per cent							
	Clinker No. 16823		Clinker No. 16843		Clinker No. 16890		Clinker No. 16839	
	$3CaO \cdot Al_2O_3 = 12.5$ $Na_2O = 0.05$ $K_2O = 0.25$		$3CaO \cdot Al_2O_3 = 11.0$ $Na_2O = 1.03$ $K_2O = 0.06$		$3CaO \cdot Al_2O_3 = 5.1$ $Na_2O = 0.32$ $K_2O = 0.02$		$3CaO \cdot Al_2O_3 = 6.2$ $Na_2O = 1.01$ $K_2O = 0.10$	
	7 days	3 months	7 days	3 months	7 days	3 months	7 days	3 months
1.0	0.047	0.093			0.054	0.110		
1.5	0.044	0.090	0.061	0.144	0.040	0.091	0.083	0.166
1.9	0.033	0.077	0.060	0.133	0.035	0.087	0.068	0.140
2.4	0.027	0.071	0.049	0.112	0.035	0.083	0.053	0.124
3.0	0.027	0.071	0.040	0.091	0.035	0.085	0.041	0.112
3.5	0.026	0.068	0.038	0.084	0.043	0.090	0.041	0.106
4.0	0.028	0.067	0.038	0.082	0.044	0.103	0.029	0.086
4.5			0.036	0.082			0.031	0.086

The Influence of Gypsum on Contraction:

The mortar prisms for contraction in air were cured in the molds 1 day, in water 6 days, and then continuously in air at approximately 75 F. and 50 per cent relative humidity. Contraction measurements are recorded as the percentage contraction calculated from the length at the time the specimens were placed in air. The results are shown in Table XII and in Fig. 17.

The general characteristics of the influence of gypsum on contraction is that the addition of gypsum decreases the contraction. However, the quantity of gypsum required to obtain the lowest contraction varies with different clinker compositions. The results show that the alkalies as well as the $3CaO \cdot Al_2O_3$ influence the gypsum requirements of the cements. For cements of low alkali content those of high $3CaO \cdot Al_2O_3$ content require larger additions of gypsum to give the lowest contraction than those of low $3CaO \cdot Al_2O_3$ content. For cements of the same $3CaO \cdot Al_2O_3$ content those of high alkali content require larger additions of gypsum than those of low alkali content. These observations are illustrated by the contraction data in Table XIII taken from the more complete data given in Table XII.

For cements of low alkali content, clinker No. 16823 with 12.5 per cent $3CaO \cdot Al_2O_3$ shows the least contraction with 2.4 to 4.0 per cent SO_3, whereas clinker No. 16890 with 5.1 per cent $3CaO \cdot Al_2O_3$ shows the least contraction with 1.9 to 3.0 per cent SO_3. On the

TABLE XIV.—MISCELLANEOUS TESTS OF CEMENTS GROUND WITH DIFFERENT PROPORTIONS OF GYPSUM—SERIES 287.

SO$_3$ Content, per cent	Specific Surface			Normal Consistency, per cent	Time of Setting				Expansion During Autoclave Tests, per cent	
	Turbidimeter, sq. cm. per g.	Air Permeability, sq. cm. pre g.	Ratio, Air to Turbidimeter		Vicat Needle		Gillmore Needle		One 5-hr. Cycle	Two 5-hr. Cycles
					Initial, hr.:min.	Final, hr.:min.	Initial, hr.:min.	Final, hr.:min.		
CLINKERS OF HIGH $3CaO \cdot Al_2O_3$ AND LOW ALKALI CONTENT Clinker No. 15367, C$_3$A, 14.3; Na$_2$O, 0.16; K$_2$O, 0.17 per cent										
1.5	1850	3210	1.74	24.0	2:35	4:40	3:15	4:50	0.127	0.167
1.9	1870	3330	1.78	24.0	2:35	6:00	3:30	6:15	....	....
2.4	1890	3460	1.83	24.5	2:30	6:00	3:30	6:15	0.058	0.097
3.0	1940	3780	1.95	24.0	2:50	6:00	3:25	6:30	....	....
3.5	2000	3980	1.98	24.0	2:50	6:10	3:20	6:30	....	....
5.0	2070	4850	2.34	23.0	....	....	....	....	0.096	0.142
Clinker No. 16823, C$_3$A, 12.0; Na$_2$O, 0.05; K$_2$O, 0.25 per cent										
1.0	1870	3280	1.75	25.0	2:15	9:50	4:00	9:50	0.190	....
1.5	1900	3440	1.81	25.0	2:05	6:00	3:30	6:00	0.246	....
1.9	1930	3560	1.84	25.0	2:00	5:30	3:30	5:30	0.222	....
2.4	1940	3790	1.95	25.5	2:40	6:10	4:00	6:10	0.189	....
3.0	1950	3970	2.04	25.5	2:35	6:25	4:10	6:25	0.219	....
3.5	1960	4160	2.12	25.5	2:40	6:40	4:30	6:40	0.241	....
4.0	1970	4320	2.19	25.5	3:00	6:35	4:20	6:35	0.234	....
CLINKERS OF HIGH OR MODERATELY HIGH $3CaO \cdot Al_2O_3$ AND HIGH OR MODERATELY HIGH ALKALI CONTENT Clinker No. 15900, C$_3$A, 13.1; Na$_2$O, 0.36; K$_2$O, 0.50 per cent										
1.5	1880	3290	1.75	24.5	2:00	4:10	3:00	4:25	0.568	....
1.9	1910	3440	1.80	24.0	2:20	5:25	3:20	5:30	....	....
2.4	1930	3640	1.88	24.0	2:20	5:30	3:30	5:45	0.380	....
3.0	1950	3870	1.98	24.0	2:50	5:20	4:05	5:45	....	....
3.5	1970	4050	2.06	24.0	3:00	5:00	4:40	5:25	....	....
5.0	2010	4650	2.25	23.0	....	....	....	....	0.649	....
Clinker No. 15498, C$_3$A, 11.2; Na$_2$O, 0.31; K$_2$O, 0.42 per cent										
1.0	1840	3200	1.74	24.5	2:35	6:15	3:30	6:30	0.530	....
1.5	1860	3270	1.76	24.5	2:30	5:45	3:30	6:00	....	....
1.9	1870	3360	1.80	24.5	2:20	5:45	3:30	6:00	....	....
2.4	1900	3580	1.88	24.5	2:20	5:45	3:30	6:00	0.320	....
3.0	1940	3810	1.97	24.0	2:10	5:30	3:30	5:45	....	....
5.0	2020	4580	2.24	23.5	....	....	....	....	0.430	....
Clinker No. 16843, C$_3$A, 11.0; Na$_2$O, 1.03; K$_2$O, 0.06 per cent										
1.5	1860	3350	1.80	25.0	0:40	2:10	1:00	3:10	0.112	....
1.9	1890	3500	1.85	25.0	1:50	4:30	3:20	4:30	0.092	....
2.4	1890	3700	1.96	25.0	2:00	4:30	3:30	4:30	0.066	....
3.0	1890	3930	2.08	25.0	2:10	5:15	3:40	5:15	0.018	....
3.5	1900	4100	2.16	25.0	2:10	5:00	3:45	5:00	0.008	....
4.0	1900	4280	2.25	25.0	2:15	4:40	3:40	4:40	0.014	....
4.5	1900	4460	2.35	25.0	2:10	5:00	3:15	5:00	0.006	....
Clinker No. 15699, C$_3$A, 10.1; Na$_2$O, 1.17; K$_2$O, 0.46 per cent										
1.0	1840	3220	1.75	23.5	1:20	3:40	2:00	4:00	0.187	....
1.5	1850	3390	1.82	23.5	1:15	3:40	2:20	4:00	....	....
1.9	1860	3510	1.88	23.5	1:35	4:00	2:40	4:15	....	....
2.4	1900	3680	1.93	23.5	1:40	4:15	2:50	4:25	0.141	....
3.0	1940	3830	1.97	23.5	1:45	4:20	3:00	4:30	....	....
5.0	2050	4400	2.15	....	....	....	....	....	....	....
Clinker No. 16827, C$_3$A, 8.1; Na$_2$O, 0.04; K$_2$O, 1.37 per cent										
1.0	1860	3220	1.73	27.0	0:20	1:30	0:30	1:50	0.085	....
1.5	1900	3350	1.76	25.0	2:00	3:40	2:30	3:50	0.101	....
1.9	1940	3450	1.78	25.5	2:40	6:00	3:20	6:00	0.101	....
2.4	1950	3650	1.87	25.5	2:25	5:50	3:40	5:50	0.085	....
3.0	1960	3890	1.98	25.0	2:50	4:30	3:45	4:50	0.082	....
3.5	1970	4070	2.07	25.0	2:40	6:30	3:40	6:30	0.077	....
4.0	1980	4250	2.15	25.0	2:30	5:40	3:35	5:40	0.071	....

TABLE XIV.—*Continued*

SO_3 Content, per cent	Specific Surface			Normal Consistency, per cent	Time of Setting				Expansion During Autoclave Test, per cent	
	Turbidimeter, sq. cm. per g.	Air Permeability, sq. cm. per g.	Ratio, Air to Turb.		Vicat Needle		Gillmore Needle		One 5-hr. Cycle	Two 5-hr. Cycles
					Initial, hr.:min.	Final, hr.:min.	Initial, hr.:min.	Final, hr.:min.		
CLINKERS OF LOW $3CaO \cdot Al_2O_3$ AND LOW ALKALI CONTENT Clinker No. 16890, C_3A, 5.1; Na_2O, 0.32; K_2O, 0.02 per cent										
1.0	1870	2960	1.58	23.0	3:40	7:40	4:00	7:40	0.010	....
1.5	1890	3090	1.63	23.5	3:50	7:20	4:00	7:20	−0.010	....
1.9	1900	3190	1.68	23.0	3:40	7:45	4:00	7:45	−0.023	....
2.4	1920	3410	1.78	23.0	2:40	5:20	4:00	5:20	−0.029	....
3.0	1950	3640	1.87	23.0	3:30	7:00	4:30	7:00	−0.031	....
3.5	1980	3870	1.95	23.0	3:30	6:40	4:30	6:40	−0.031	....
4.0	2000	4060	2.03	23.5	3:20	6:50	4:30	6:50	−0.029	....
Clinker No. 15623, C_3A, 4.8; Na_2O, 0.05; K_2O, 0.18 per cent										
0.03	1800	2860	1.59	23.0	8:20	12:00	9:20	13:00	0.005	0.007
1.0	1840	3120	1.70	22.0	3:15	6:00	4:10	6:00	....	....
1.5	1860	3260	1.76	22.0	3:15	5:50	4:30	6:00	....	....
1.9	1880	3410	1.81	22.0	3:00	5:45	4:20	5:50	....	....
2.4	1910	3620	1.90	22.0	2:50	5:40	4:00	5:40	−0.045	−0.042
Clinker No. 15670, C_3A, 2.4; Na_2O, 0.05; K_2O, 0.23 per cent										
0.17	1800	2630	1.46	20.0	8:20	12:00	9:00	13:00	0.033	0.038
1.0	1870	2920	1.56	19.5	5:00	8:50	5:15	8:50	....	....
1.5	1910	3080	1.62	20.0	5:25	8:45	5:50	8:45	....	....
1.9	1950	3220	1.65	20.0	5:10	8:40	5:30	8:40	....	....
2.4	1970	3390	1.72	20.0	5:00	8:30	5:00	8:40	−0.017	−0.017
CLINKERS OF LOW $3CaO \cdot Al_2O_3$ AND HIGH ALKALI CONTENT Clinker No. 16839, C_3A, 6.2; Na_2O, 1.01; K_2O, 0.10 per cent										
1.5	1890	3190	1.69	24.0	0:30	4:15	1:50	4:15	0.032	....
1.9	1910	3380	1.77	24.0	2:15	5:00	2:50	5:00	0.032	....
2.4	1940	3600	1.86	24.5	2:05	5:40	3:40	5:40	0.013	....
3.0	1980	3890	1.96	25.0	2:15	6:20	4:00	6:20	−0.007	....
3.5	2010	4050	2.01	25.5	2:30	5:35	3:40	5:35	−0.021	....
4.0	2030	4250	2.09	25.5	1:45	5:15	3:30	5:15	−0.017	....
4.5	2060	4460	2.17	25.5	2:10	5:20	3:30	5:20	−0.009	....
Clinker No. 16831, C_3A, 5.7; Na_2O, 0.05; K_2O, 1.37 per cent										
1.0	1840	3510	1.91	26.0	3:15	5:00	3:45	5:00	0.102	....
1.5	1820	3580	1.97	24.5	3:30	5:30	3:45	5:30	0.090	....
1.9	1810	3630	2.01	24.0	3:00	6:10	4:30	6:10	0.074	....
2.4	1820	3820	2.10	23.5	3:15	6:20	4:30	6:20	0.056	....
3.0	1830	4040	2.21	23.5	3:15	6:20	4:30	6:20	0.044	....
3.5	1850	4220	2.28	23.0	3:00	6:00	4:00	6:00	0.038	....
4.0	1860	4400	2.37	23.0	2:45	5:50	4:00	5:50	0.031	....

other hand, for cement of high $3CaO \cdot Al_2O_3$ content the clinker with low alkalies, No. 16823, shows the least contraction with 2.4 to 4.0 per cent SO_3, whereas the clinker with high alkalies, No. 16843, shows the least contraction with 3.5 to 4.5 per cent SO_3. Likewise, for cements of low $3CaO \cdot Al_2O_3$ content the clinker with low alkalies, No. 16890, shows the least contraction with 1.9 to 3.0 per cent SO_3, and the clinker with high alkalies, No. 16839, shows the least contraction with 4.0 to 4.5 per cent SO_3.

Figure 19 shows that the contractions of the different cements are very nearly equalized by the use of proper amounts of gypsum. When the cements are compared on the basis of an SO_3 content of 1.9 per cent, the different cements show contractions ranging from 0.025 to 0.068 per cent at 7 days and 0.060 to 0.148 per cent at 3 months—spread of 0.043 and

0.088 percentage points, respectively. When the same cements are compared on the basis of the amount of gypsum that gives the least contraction the values range from 0.019 to 0.040 per cent at 7 days and from 0.060 to 0.093 per cent at 3 months—here the spread is reduced to 0.021 and 0.033 percentage points at the respective ages.

Thus, the results indicate that, for cement of high $3CaO \cdot Al_2O_3$ regardless of their alkali content or cements high in alkalies regardless of their $3CaO \cdot Al_2O_3$ content, the contraction on drying can be decreased as much as 30 to 50 per cent by the use of larger additions of gypsum than are permitted by the current A.S.T.M. Specifications C 150 – 44.[3] For cements of low $3CaO \cdot Al_2O_3$ and low alkali content the contraction would not be decreased by larger additions of gypsum. For each clinker the SO_3 content which gives the lowest contraction in air is likewise the SO_3 content which gives the highest strength.

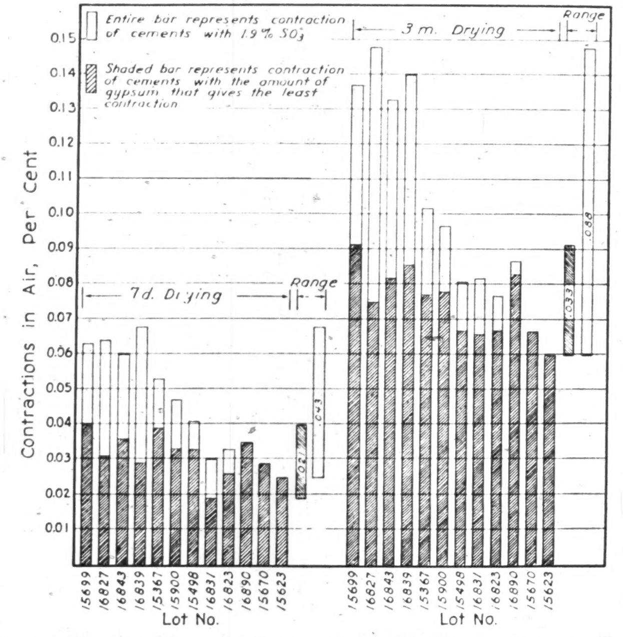


FIG. 19.—Diagram Showing How the Contractions of Different Cements Tend to Be Equalized by the Use of Proper Amounts of Gypsum.

Miscellaneous Tests of the Cements:

The results of miscellaneous tests of the cements are given in Table XIV. For cements prepared from the different clinkers there is considerable difference in the water required for normal consistency. The cements of high $3CaO \cdot Al_2O_3$ content require more water than do the cements of low $3CaO \cdot Al_2O_3$ content. For cements prepared from any one clinker, with SO_3 content varied, the water required for normal consistency does not vary appreciably except that the cement with 5.0 per cent SO_3 requires less water than do the corresponding cements of lower SO_3 content.

As the SO_3 content is increased, the time of set is generally increased for cements of high $3CaO \cdot Al_2O_3$ content and decreased for cements of low $3CaO \cdot Al_2O_3$ content. All cements meet the requirements of the A.S.T.M. Methods C 191 - 44[6] for time of set except the clinkers of low $3CaO \cdot Al_2O_3$, (Nos. 15623 and 15670) when tested without added gypsum. Under this condition the cements are very slow setting. All cements pass the pat test for soundness. Increasing the SO_3 content does not greatly alter the expansion in the autoclave test though there is a general trend of slightly lower expansions with increasing SO_3.

The specific surface was determined by the Wagner turbidimeter and by the air permeability method (22, 23). The results obtained by these methods are given in Table XIV. It is shown that for each clinker the ratio of the specific surfaces determined by the air permeability method to those determined by the Wagner method increases progressively with increasing SO_3 content. It is probable that such variations in specific surface by the two methods can be accounted for on the basis that the Wagner method does not adequately account for all of the surface of the minus 7.5 μ particles. Thus, since a large percentage of the gypsum is present as $-$ 7.5 μ particles, the ratio of the specific surfaces determined by the air permeability method to those determined by the Wagner method would increase with larger additions of gypsum.

TABLE XV.—OPTIMUM SO_3 CONTENT OF CLINKERS OF DIFFERENT COMPOSITION.

Clinker	C_3A	Na_2O	K_2O	For Proper Retardation (Heat Curves)	For Highest Strength	For Lowest Contraction	To Avoid Abnormal Expansion	Optimum SO_3, per cent
Clinkers of High $3CaO \cdot Al_2O_3$ and Low Alkali Content								
No. 15367	14.3	0.17	0.16	3.0	2.4 to 3.0	3.0	3.5	3.0
No. 16823	12.5	0.05	0.25	3.5	2.4 to 3.0	2.4 to 3.0	>4.0	3.0
Clinkers of High or Moderately High $3CaO \cdot Al_2O_3$ and High or Moderately High Alkali Content								
No. 15900	13.1	0.36	0.50	4.0	3.5	3.5	5.0	3.5
No. 15498	11.2	0.31	0.42	3.0	2.4 to 3.0	2.4 to 3.0	>3.5	3.0
No. 16843	11.0	1.03	0.06	4.0	3.5 to 4.5	3.5 to 4.5	>4.5	4.0
No. 15699	10.1	1.17	0.46	4.5	5.0	5.0	5.0	4.5
No. 16827	8.1	0.04	1.37	4.0	3.0 to 4.0	3.5 to 4.5	>4.0	3.5
Clinkers of Low $3CaO \cdot Al_2O_3$ and Low Alkali Content								
No. 16890	5.1	0.32	0.02	1.9	1.9 to 2.4	1.9 to 3.0	3.0	1.9
No. 15623	4.8	0.05	0.18	1.9	1.5 to 2.4	1.5 to 1.9	>2.4	1.9
No. 15670	2.4	0.05	0.23	1.9	1.5 to 1.9	1.5 to 1.9	>2.4	1.9
Clinkers of Low $3CaO \cdot Al_2O_3$ and High Alkali Content								
No. 16839	6.2	1.01	0.10	4.5	3.5 to 4.5	4.0 to 4.5	>4.5	4.0
No. 16831	5.7	0.05	1.37	3.0 to 3.5	3.0 to 3.5	2.4 to 3.0	>4.0	3.0

The Optimum SO_3 Content for the Different Clinkers:

In the studies described in this report four methods of test have been used to determine the optimum SO_3 content for the different clinkers. The rate of heat-liberation curves were used to determine the minimum SO_3 content required to give the type of curve that would meet the definition of a properly retarded cement, strength tests were made to determine the SO_3 content required for the best strengths, contraction measurements were made to determine the SO_3 content required for the lowest contraction, and expansion measurements were made to detect the occurrence of abnormal expansion resulting from the use of larger additions of gypsum. The results of these tests are summarized in the Table XV. It is seen that the SO_3 values by the different methods are in relatively good agreement. A suggested value for an optimum percentage is shown which is not higher than that indicated by any of the four methods.

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[6] Methods of Test for Time of Setting of Hydraulic Cement by the Vicat or Gillmore Needles (C 191 - 44), 1944 Book of A.S.T.M. Standards, Part II, p. 66.

These data show that results obtained by calorimetric methods, and the tests for strength, contraction and expansion are all in very good agreement in showing the optimum SO_3 content for the different clinkers. The relationship between the SO_3 contents required for the highest strength and lowest contraction is shown also in Fig. 17.

Previous investigators (**24, 25, 26**) have studied the influence of cement composition on the contraction with the SO_3 content of the cements maintained constant at 1.8 to 1.9 per cent. From the results of such investigations it has been concluded that the $3CaO \cdot Al_2O_3$ has the greatest influence on contraction and the contraction of hardened cement pastes stored in air increases with increasing $3CaO \cdot Al_2O_3$ content. The results of the present investigation show that the high coefficients of contraction of the $3CaO \cdot Al_2O_3$ and alkalies can be very considerably reduced and that the contraction of different cement compositions can be equalized, or very nearly equalized, by a proper adjustment of the gypsum content.

SUMMARY AND CONCLUSIONS

Twelve commercial clinkers, representing the range of chemical composition found in portland cements, were ground in a laboratory mill with various additions of gypsum. The resulting cements were used to study the influence of gypsum on the hydration and properties of portland-cement pastes. Five of the clinkers were ground in commercial mills with approximately 1.8 per cent SO_3 to three widely different specific surfaces. The latter cements were used to study the influence of fineness upon the rate of hydration with SO_3 constant.

A conduction calorimeter was used to determine the rate of hydration of neat cement pastes, mortar prisms were used to determine the physical properties of the hardened paste.

A properly retarded cement is defined on the basis of the shape of the heat-liberation curve during the first 30 hr. of hydration. A properly retarded cement is one that contains the minimum quantity of gypsum required to give a heat-liberation curve that shows two cycles of ascending and descending rates and that shows no appreciable change with larger additions of gypsum. The results of the physical tests show that when considering cements ground from a given clinker, those containing the proper amount of gypsum to give this type of curve will develop the highest strength and the lowest contraction.

The quantity of gypsum required to obtain a properly retarded cement varies with the composition and fineness of the cement. The results show that the alkalies as well as the $3CaO \cdot Al_2O_3$ content influence the gypsum requirements of the cements. With cements of low alkali content those of high $3CaO \cdot Al_2O_3$ content require larger additions of gypsum than those of low $3CaO \cdot Al_2O_3$. For cements of the same $3CaO \cdot Al_2O_3$ content those high in alkalies react with gypsum more rapidly and require larger additions of gypsum than those low in alkalies. There is some evidence that cements containing Na_2O require larger amounts of gypsum than do similar cements containing an equivalent quantity of K_2O.

Gypsum retards the early hydration of cements of high or moderately high

$3CaO \cdot Al_2O_3$ content and accelerates the hydration of cements of low $3CaO \cdot Al_2O_3$.

Without added gypsum, cements of high or moderately high $3CaO \cdot Al_2O_3$ regardless of their alkali content and cements of low $3CaO \cdot Al_2O_3$ that are high in alkalies react with water so rapidly that an immediate flash-set occurs. This phenomenon is the result of the rapid solution of the anhydrous aluminate phases and the rapid crystallization of hydrated calcium aluminates. With added gypsum, the gypsum dissolves in the mixing water together with lime formed by the hydrolysis of compounds present in the cement. The saturated lime-gypsum solution depresses the solubility of alumina in the aqueous solution and thereby retards the hydration of the aluminate phases.

In the presence of the saturated lime-gypsum solution the aluminate phases dissolve at a retarded rate and react with the lime and gypsum to form an insoluble calcium sulfoaluminate. By this process the gypsum may eventually become depleted and its concentration in the aqueous solution decreased. If at this time there is still present a quantity of unhydrated aluminate phases, a rapid reaction will ocur. Such a rapid reaction appears to be the same as that which occurs in the immediate flash-set, that is, a rapid solution of the anhydrous aluminate-phases and a rapid crystallization of hydrated calcium aluminates. With larger additions of gypsum this rapid reaction is eliminated and larger amounts of calcium sulfoaluminate are formed in place of hydrated calcium aluminates.

It appears that at least part of the alkalies of the cement are present in the aluminate phases, and that aluminate phases containing alkalies react with water more rapidly than do similar phases which are alkali-free or of lower alkali content. Thus the cements of higher alkali content require larger additions of gypsum for proper retardation than do similar cements of lower alkali content.

Increasing the specific surface of the cement increases the quantity of aluminate phases available for reaction with water at early ages and thereby increases the quantity of gypsum required for proper retardation of cements of moderately high or high $3CaO \cdot Al_2O_3$ content.

Cements of low $3CaO \cdot Al_2O_3$ and low alkali content without added gypsum can be mixed with water without the occurrence of a flash-set. With cements of this type an amorphous hydrated calcium ferrite precipitates on the surface of the cement particles and seals the surface in a manner such as to retard subsequent hydration. With added gypsum a crystalline hydrated calcium sulfoferrite is formed which does not seal the surface and the hydration is accelerated.

The results show that varying the gypsum content of the cement will alter the rate of hydration at early ages and alter the hydration products that are formed. It seems probable that such changes in the hydration process would alter the structure of the hardened paste. No attempt was made to examine the structure of the paste by direct methods. However, the results of physical tests indicate that the structure of the hardened paste is altered.

The results of the physical tests show that for many cements the strengths can be increased and the contraction on drying or the expansion in water storage decreased by the use of larger additions of gypsum than are permitted by current specifications. In some instances in the present investigation the strengths were increased by as much as 20 to 50 per cent and the contraction decreased by as much as 30 to 50 per cent. For cements of low $3CaO \cdot Al_2O_3$ and low alkali content the strengths were not increased nor was the contraction decreased by larger additions of gypsum. The cements high in $3CaO \cdot Al_2O_3$ regardless of their alkali content or cements high in alkalies regardless of $3CaO \cdot Al_2O_3$ content require the larger additions of gypsum.

Gypsum could be added in larger amounts than is permitted by present specifications without danger of delayed expansion.

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DISCUSSION

MR. BENJAMIN WILK.[1]—What would be the effect on the hardening of concrete, if it were cured at higher temperatures?

MR. WILLIAM LERCH (*author*).—The tests described in this report were made at a temperature of 75 F. Some additional tests have been made to study the effect of temperature on the rate of hydration. Higher temperatures have an effect approximately similar to that of increasing the fineness; that is, the hydration is accelerated.

MR. G. L. KALOUSEK[2] (*by letter*).—The results presented by the author on the effect of gypsum on properties of hydrating cements and concretes comprise a distinctive contribution to the knowledge of cement technology. The paper presents information long needed to answer many questions regarding the correct amounts of gypsum required for retardation or acceleration of the reactions of setting and hardening.

The twelve cements used in the tests give a good representation of composition and alkali contents among commercial cements. However, because of the peculiarities in behavior of many cements due to unknown factors, the proper choice of "representative" cements is not always realized. It is not the intent to criticize the report on the ground that too few cements were used or the data are limited; the data for most tests are given in large numbers. It is felt, nonetheless, that the inclusion of certain other cements may have altered some of the interpretation made of the data.

Principally on the basis of the thermal behavior of the cements during hydration, the author advances a theory to explain the mechanism of setting and hardening as related to the alumina-bearing phase, tricalcium aluminate. This theory appears inadequate to account satisfactorily for many data published by other investigators. It is true that such data probably in most instances were obtained on cements differing in many respects from those of the author. However, a theory to be general should be applicable to all cements. It is with this thought in mind that this discussion is presented.

Lea[3] in commenting on the theories of setting of cements states, "it cannot be said that at the present time any definite decision as to the correctness of these various theories can be made as it seems not unlikely that certain elements of the whole truth reside in many of them." One of these "elements of truth," as it would appear to be from an abundance of prevailing data, observations, and deductions, is the generally accepted hypothesis of the formation of a slightly permeable hydrous film on the surface of cement grains to retard the reactions of setting and hardening. The author discards this concept in explaining the retardation of setting by gypsum, stating that it is due to the decreased solubility of the anhydrous aluminates in presence of $CaSO_4$ in solution. The cause of the appearance of the third maximum in his rate-of-heat – evolution-time curves he ascribes to an acceleration of the hydration of the alumina phase which results from an increase in solubility of the aluminate upon depletion of gypsum in solution. Reasons are advanced in the following paragraphs why it is believed that such a theory is less tenable than one based on the formation of semipermeable membranes of probably a metastable nature.

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[1] Standard Building Products Co., Detroit, Mich.

[2] Chemist, Research Dept., Owens-Illinois Glass Co., Toledo, Ohio.

[3] F. M. Lea and C. H. Desch, "Chemistry of Cement and Concrete," Edward Arnold and Co. (London), (1935).

The author does not report any values for the concentrations of alumina in the aqueous phase of his cement pastes. Since such values have a pertinent bearing on the theory, some of the previously published data will be considered briefly. Attention has to be called first to the fact that the anhydrous phases as such have no true solubility in water because they are decomposed; hence, the solubility of the products (hydrates) formed is in reality the determining factor when discussing the solubility of the anhydrous phase. The author's statement that calcium sulfoaluminates (phases formed in presence of soluble sulfates) are less soluble than the hydrated calcium aluminates (phases formed in presence of $Ca(OH)_2$) is correct, but the amounts in solution are very small for both types of salts. The writer[4] has reported values of about 0.0002 g. per liter of Al_2O_3 as the solubility of the sulfoaluminates in saturated or nearly saturated lime-gypsum solutions. The value for the hydrated calcium aluminates has variously been reported from 0.0004 to 0.004 g. per liter of Al_2O_3 in saturated or nearly saturated lime solution. These concentrations, however, are so markedly smaller than those of the other constituents in solution that, from the standpoint of theories of solution, it would be difficult to differentiate among the values given or to ascribe any real meaning to the small difference observed. Inasmuch as the alkalies have an effect on the solubility of the hydrous aluminates and sulfoaluminates,[4] it might seem that in the aqueous phase of hydrating cements a significant difference might be found in the solubility of the phases mentioned. Examination of extensive data published by the writer jointly with others[5] on the compositions of aqueous extracts from cement clinkers, showed that within limits of experimental errors, and making allowance for any Cr_2O_3 present, no significant difference could be found in extracts obtained from pastes, with and without additions of gypsum, filtered immediately after mixing and 2 hr. after mixing. The values, ranging between 0.002 to 0.010 g. per liter of Al_2O_3, are very small compared to the concentrations of the other constituents.

The author in explaining the heat of immediate hydration, the first maximum in the heat curves, states that the mixing water during about the first 5 min. is not saturated with lime and gypsum, and that during this period the anhydrous aluminates are being dissolved and the hydrated calcium aluminate is being precipitated. This claim for the rate of solution is not borne out by the data of other investigators. Forsen[6] has shown that gypsum dissolves rapidly, and also that the solutions from cement pastes are almost instantly saturated or supersaturated with $Ca(OH)_2$ and saturated with respect to gypsum. Data obtained by the writer on the solution of

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[4] G. L. Kalousek, "Study of a Portion of the System $CaO-Al_2O_3-SO_3-Na_2O-H_2O$ at 25 C.," Dissertation, University of Maryland, College Park, Md. (1941).

[5] G. L. Kalousek, C. H. Jumper, J. J. Tregoning, "Composition and Physical Properties of Aqueous Extracts from Portland Cement Clinker Pastes Containing Added Materials," *Journal of Research*, Nat. Bur. Standards, Vol. 30, p. 215 (1943).

[6] L. Forsen, "The Chemistry of Retarders and Accelerators," Symposium on the Chemistry of Cements (Stockholm), p. 298 (1938).

$Ca(OH)_2$ in the mixing water agreed with that of Forsen. Although such results would contradict sharply the author's contention on the rates of solution during the first few minutes, it should be pointed out that cements which have been in storage a long time and are partially hydrated might show a slower rate of solution of $Ca(OH)_2$. Because gypsum dissolves rapidly it would be available for sulfoaluminate formation, and it is highly probable that calcium sulfoaluminate is precipitated instead of a calcium aluminate hydrate, immediately upon the addition of the mixing water. This statement is based on tests and observations by Forsen,[6] results of comprehensive studies by Jones[7] on systems involving $CaO\text{-}Al_2O_3\text{-}SO_3\text{-}Na_2O\text{-}K_2O\text{-}H_2O$, and by results of the writer[4].

In order to discuss the postulate of the formation of protective films and contrast it with the author's explanation, a brief résumé is given of some of the theories advanced. Forsen[6] presented an extensive amount of experimental data to support the hypothesis that a film of a sulfoaluminate, specifically the monosulfate form of the salt, is formed and retards the reaction of the hydration of the aluminous phases. Jones[7] in a recapitulation of his studies likewise favors the formation of a film of sulfoaluminate, but he considers that the solid solution of the sulfoaluminate is the product formed. Roller[8] expressed the belief that a film of tetracalcium aluminate hydrate was formed even in the presence of gypsum in solution. As already stated, it appears doubtful that hydrated calcium aluminate precipitates in the presence of sulfate in solution.

In what manner the deposit of sulfoaluminate as a film may retard the hydration of the anhydrous alumina phases is not known, especially in cements where the presence of silicates may play a part. Thus, additions of alkali sulfate to cement clinkers,[9] or the K_2SO_4 already present in some clinkers[10,11], do not retard the reactions of hydration even though sulfoaluminates are being formed[11]. Actually alkali salts[9] and hydroxides[3] accelerate the reactions of setting. Evidently certain conditions favoring the stability of the film must prevail. Roller[8] pointed out that $Ca(OH)_2$ must be present in solution to stabilize the film, Forsen[6] called attention to the fact that a large decrease in $Ca(OH)_2$ in solution is caused by NaOH and KOH of even moderate concentration, and this confirms Roller's finding.

The preceding considerations would permit the deduction that the nature of the aqueous phase has a marked influence on the course of the reaction of hydration. It should follow that as the composition of the solutions changes, a change occurs in the nature of the solid products. In studies of systems pertaining to cements, the writer[12] has shown that even relatively low concentrations of NaOH (5 g. per liter as Na_2O) decrease markedly the lime content of the hydrous lime silicate gel formed compared to the composition found without the NaOH.

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[7] F. E. Jones, "The Formation of the Sulfoaluminates and Sulfoferrites of Calcium in the Portland Cement-Water System," *Journal of Physical Chemistry*, Vol. 49, No. 4, p. 344 (1945).

[8] P. S. Roller, "The Setting of Portland Cement," *Industrial and Engineering Chemistry*, Vol. 26, p. 669 (1934).

[9] C. H. Jumper and G. L. Kalousek, "Effect of Admixtures on Temperature Rise of Cement Pastes," *Rock Products*, Vol. 54, April, May, and June, 1942.

[10] W. C. Taylor, "Further Phase-Equilibrium Studies Involving the Potash Compounds of Portland Cement," *Journal of Research*, Nat. Bur. Standards, Vol. 29, p. 437 (1942).

[11] G. L. Kalousek, C. H. Jumper, and J. J. Tregoning, "Potassium Sulfate in Cement Clinkers," *Rock Products*, April, 1941.

[12] G. L. Kalousek, "Studies of Portions of the Quaternary System Soda-Lime-Silica-Water at 25 C.," *Journal of Research*, Nat. Bur. Standards, Vol. 32, p. 285 (1944).

Also it was found that NaOH in moderate concentration results in the formation of the solid solution form of the sulfoaluminates. Direct evidence is not yet available as to the alterations that may take place in the composition of the hydrous film in cements. However, it is likely that as the composition of the solution is changed there results a change in the film. Certain deductions by inference regarding these changes may be drawn from the heat curves for the hydration of $3CaO \cdot SiO_2$ without any additions. The author has mentioned that this compound in reacting with water gives a rate-of-heat-liberation curve similar to that of cements of low $3CaO \cdot Al_2O_3$ and low alkali. That is, there is first a rapid evolution of heat during the first few minutes, termed the heat of immediate hydration, followed by much lower rate for a few hours, and then there occurs a more rapid evolution of heat for a while. The explanation of these variations in the rate of heat liberation may be according to the following reasons. During the first few minutes the reaction is rapid and a deposit of a lime silicate is formed on the surface of the crystals. The gel first formed must contain less lime than that formed later because initially a part of the lime is required to saturate the solution. After the solution is saturated, in a few seconds for pastes of normal consistency, the gel subsequently formed is richer in lime. Why the film deposited retards the hydration for a while, as indicated by the slower rate of heat evolution, is not known. It must be more impervious to the water than that deposited later, but upon changing over to a product of different composition it becomes more pervious to the water and the rate of hydration is increased.

A rate-of-heat-liberation curve for $3CaO \cdot Al_2O_3$ cannot be determined as for $3CaO \cdot SiO_2$, because of the very rapid rate at which the reaction occurs. Apparently no film is deposited. This compound when in cements shows a markedly slower rate of reaction, due probably to the effect of the other constituents in cements. The author has shown that the curve for cements of moderate or high $3CaO \cdot Al_2O_3$, depending on the amount of gypsum added, may show three maxima, the first two being similar to those for $3CaO \cdot SiO_2$ except they are higher and the second occurs sooner. As already mentioned, the author ascribed the first to the formation of hydrous calcium aluminate, but it could be due also to the formation of the sulfoaluminates. The cause for the second maximum is not given. The third maximum occurs if insufficient gypsum had been added and is explained as being the result of the rapid hydration of the remaining $3CaO \cdot Al_2O_3$ above that required to combine with all the sulfate. The author could not account for the third maximum if the hypothesis of the film formation were adopted. The following statement from his report summarizes the ground for the rejection of the film hypothesis: "If this decrease in the rate of heat liberation were to be explained entirely on the basis of the formation of a protective film of hydration products it would be difficult to explain the subsequent disappearance of the protective film, as would be necessary, to permit the rapid reaction and high rate of heat liberation."

Any explanation of the third maximum merits consideration of the change in the aqueous phase. Prior to the occurrence of this rapid liberation of heat, the sulfate disappears from solution and there is left essentially a solution of alkali hydroxides for cement of even moderate alkali contents. This change in solution (from calcium and alkali sulfates and hydroxides to only alkali hydroxides with little lime) could result in a chemical as well as physical change in the reaction products leading to an increased permeability of the film. Not only would the reaction be accelerated by the availability of more water to the anhydrous particles, but as already mentioned the reactions of hydration of cements are accelerated by addition of alkali salts.

The explanation of the second maximum in the heat-liberation curves would appear to deserve more consideration than given it. Neither the hydration of $3CaO\cdot Al_2O_3$ alone, nor $3CaO\cdot SiO_2$ alone, accounts for this maximum. It does not seem unlikely that both these phases are hydrating simultaneously. It is possible that the hydration of the siliceous phases affects that of the aluminous phases and conversely the hydration of the siliceous phases may be altered by the reactions of the aluminous phases. This point is of interest in connection with the author's claim that the alkalies may be associated with the aluminous phases and that these alkali-containing phases, as indicated by heat-liberation characteristics, react more rapidly with gypsum than do similar phases free of alkalies. One may raise the question whether the alkalies could not originate equally as well from the siliceous phases since they are probably hydrating relatively fast at the point of the second maximum. Once in solution, these would be expected to accelerate the reaction of the aluminous phases with gypsum. The author may be correct in his deductions, but it seems that too little is known about the hydrolysis of the alkali-containing lime silicates to permit any conclusion as to the origin of the alkalies. Furthermore, the presence of the alkali sulfates in clinker also may be expected to accelerate the rate of reaction.

MODULUS OF ELASTICITY OF AGGREGATES AND ITS EFFECT ON CONCRETE*

BY H. A. LARUE[1]

SYNOPSIS

Materials in the State of Missouri, available for use as concrete aggregates, show considerable variations in hardness and other properties. Some make excellent concrete, which is durable and resists weathering agencies well, while others do not give such good results.

This paper gives a report of some studies made on the properties of the aggregates and their effects on the concretes in which these aggregates were incorporated. Attention has been chiefly given to the elastic properties of the aggregates and their relations to those of the concrete. The data reported here may serve to explain in part some characteristics which concrete has shown under service conditions.

In cooperation with the Bureau of Materials of the Missouri Highway Department, the Engineering Experiment Station at the University of Missouri has carried on a series of experiments to determine the relation between the physical characteristics of certain stones and that of the concrete in which the stones are used as coarse aggregate.

This program is a continuation and extension of the research initiated by Willis and De Reus (1)[2] on aggregates from several sources in Missouri. Their work was confined to a study of the effects of fine aggregates on mortars, whereas the investigations reported here deal with the effects of coarse aggregates on concrete mixtures.

General statements have been made by other investigators to the effect that the elastic properties of concrete vary with the stiffness of the aggregates used. Results of some experiments are reported by Richard L. Humphrey (2), which included compression tests on concrete cylinders for strength and determination of modulus of elasticity, with results showing different values for the modulus for various aggregates—granite, limestone, gravel and cinders. Koenitzer (8) included the statement that: "Aggregates have an important influence on the elastic properties of the resulting concrete." Noble (7) stated that a "... very marked effect of variations in aggregates upon the value of the modulus of elasticity is particularly significant, the extent of this variation being such that it should by all means enter into the design of reinforced concrete structures." The results as reported by these authors show that hard stones, such as granite, produce a higher modulus of elasticity in the concrete than aggregates known to have lower elastic properties. But no reports on the properties of concrete show that any quantitative relations have been determined between the modulus of elasticity of concrete and the elastic properties of the stones which have been used as aggregates. However, other reports obtained for concretes made with different stones indicate that the modulus of concrete is affected very little by the several types of aggregates.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] Associate Professor of Civil Engineering, University of Missouri, Columbia, Mo.

[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 1309.

Suggestions have been made by other investigators that a definite study is needed to determine the modulus of elasticity on individual aggregates entering into concrete mixtures.

A report by Stanton Walker (6) on some investigations of the modulus of elasticity for concrete made from four different coarse aggregates: crushed limestone, pebbles, crushed blast-furnace slag, and crushed granite, states that: "These tests show *little variation* in either strength or modulus of elasticity of concrete made from the four types of coarse aggregates. This result should be contrasted with the published tests on concrete made from different aggregates. These tests undoubtedly show the correct relations."

Perhaps the lower precision of the instruments described by the authors as being used in these experiments may account for reaching this conclusion.

The data presented in the present report concern that *little variation* which apparently is shown in the modulus of elasticity of concrete, due to the use of aggregates having differing moduli of elasticity. In order to detect the small differences in the elastic properties of the various concretes, very accurate measurements of the deformation of the specimens under relatively small loads are necessary. The measurements made in this series of experiments were of a high degree of precision, observations of deformation being obtained with Huggenberger tensometers. More detailed explanation of the sensitivity of the tensometers is given later in this report.

Admitting that the differences in modulus are small for different concretes, nevertheless, they may well be of sufficient magnitude to cause unequal distribution of stress and deformation between the coarse aggregates and the mortar matrix due to expansion or contraction from temperature effects and wetting and drying. It is readily conceivable that such differentials can cause damaging effects in the internal structure of the concrete mixture, which is essentially a conglomerate, made up of constituents with differing characteristics.

A report by D. A. Abrams (5) includes a general conclusion as follows: "Recent investigations have shown that the strength and resistance to wear of concrete is dependent only to slight degree upon the weight, hardness or toughness of the material of which the coarse aggregate is composed, so long as the compressive strength of the material is not lower than that of the concretes. The high strength secured from light-weight aggregates consisting of burnt shale has shown the fallacy of the older views that the strength of the concrete is dependent upon the strength of the aggregate. More thorough investigations have shown that the concrete made from limestones, gravels, granites, traps, sandstones and blast-furnace slag, as coarse aggregates, give essentially the same strength, so long as the grading is similar and the quantity of water absorbed by the aggregates is taken into account."

Certain factors which come within the scope of this study show agreement with the general opinion given above. The specific feature that is of major importance in the acceptance of Mr. Abrams' statement pertains to the condition that the aggregate should be of a uniform character as to physical properties, and that better results with concrete will be obtained if the aggregates are of

TABLE I.—SIEVE ANALYSES OF SANDS USED.

Sieves	Total Passing, per cent
⅜-in.	100.00
No. 4	99.84
No. 8	96.30
No. 14	77.18
No. 28	48.49
No. 48	10.31
No. 100	1.92

approximately the same as or of greater hardness and toughness than the mortar in which it is embedded.

Assuming this to be a correct statement as to the strength of concrete, the studies reported herein have led to the principal purpose of determining the relationship of the modulus of the concrete to two factors only: (1) the modulus of the stones used for aggregates and (2) the modulus of the mortar in which the aggregates are embedded. Since the character of the mortar has an important bearing on the qualities of any concrete mixture, this factor is included in the development of the theory as to the relationship existing between the properties of the aggregates and those of the concrete.

TABLE II.—ELASTIC MODULUS OF LIMESTONES.

Bowling Green Limestone		Carthage Limestone				Plattin Limestone	
Specimen Number	Elastic Modulus, $E_s \times 10^{-6}$	Specimen Number	Elastic Modulus, $E_s \times 10^{-6}$	Specimen Number	Elastic Modulus, $E_s \times 10^{-6}$	Specimen Number	Elastic Modulus, $E_s \times 10^{-6}$
		HIGH MODULUS		LOW MODULUS			
C12V	2.049			D11	6.06	P4	10.30
C16V	1.362			D12	5.959	P5	11.18
C18V	2.193	B13	8.139			P7	11.18
C19V	2.138			D14	6.198	P8	10.41
C21V	1.257	B16	9.603			P9	10.41
C22V	2.043	B19	10.284			P10	11.83
C30V	1.690	B20	9.671			P11	12.08
C35V	2.175	B21	8.547			P12	12.30
C36V	1.684	B22	9.228			P13	12.00
C49V	1.825	B23	9.773			P14	11.27
		B24	8.752			P15	11.27
Not used in test		B27	8.207			P16	11.09
C10V	3.014	B28	8.479			P17	11.88
C15V	2.270			D29	6.147	P20	12.06
C28V	1.707	B30	9.399			P21	11.06
C40V$_1$	2.732	B32	8.581			P23	12.30
C40V$_2$	2.732	B33	8.343			P24	10.55
C43V	2.634	B34	9.228			P25	10.55
C12H	1.454	B35	8.309			P26	10.48
C15H	4.482	B36	11.646			P27	10.57
C16H	2.492	B37	8.207			P28	12.30
C21H	1.756	B38	10.216			P29	10.79
C28H	2.820	B39	9.296			P30	11.84
C30H	2.276	B40	8.649			P31	10.93
				D41	7.219	P32	11.88
		B42	9.024			P33	12.30
		B45	8.479				
		B46	9.228			Not used in test	
		B47	9.433			P1	9.65
		B48	9.841			P2	9.37
		B49	9.024			P3	12.45
		B50	8.479			P6	7.21
				D51	7.253	P18	8.79
		B54	10.250			P19	9.76
		B55	8.547			P22	9.76
		B56	9.671			P22H	9.23
						P26H	11.18
						P29H	11.47
		The following specimens were drilled and tested by M. E. De Reus:					
				D1	6.4	P3jc	10.6
				D3	7	P4jc	12.7
				D4	7		
				D6	6.7	Not used in test	
				D8	6.6	P1jc	9.5
				D9	7.3	P2jc	9.5
Average	1.877	Average	9.047	Average	6.733	Average	11.37
Highest value	2.193	Highest value	11.65	Highest value	7.30	Highest value	12.75
Lowest value	1.257	Lowest value	8.14	Lowest value	5.96	Lowest value	10.48

Since the reports of earlier tests to which reference has been made were based on concretes made from cements which did not have the high strengths that present-day cements exhibit, the effects of coarse aggregates on the properties of the concrete were less evident than now.

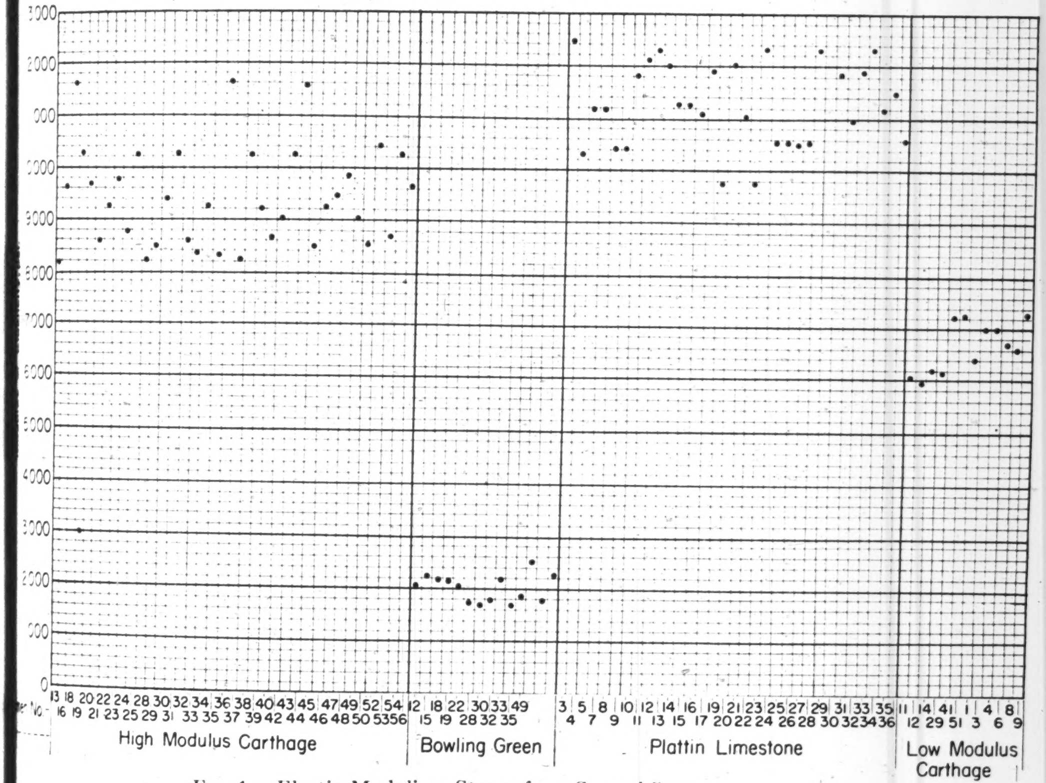

FIG. 1.—Elastic Moduli on Stones from Several Sources.

MATERIALS

The portland cement used in the manufacture of the cylinders was one of the standard brands which conformed satisfactorily to A.S.T.M. Standard Specifications for Portland Cement (C 150 - 41)[3].

The sand was obtained from the Missouri River at Jefferson City, Mo. For the purpose of obtaining uniformity of composition in the mortar, the sand was separated by screening into several fractions, and these fractions were recombined in proper proportions to get a sand of the gradation shown in Table I.

Stone for coarse aggregate was obtained from several sources in the state to represent a wide range in physical characteristics, but primarily to show distinctly different values for the modulus of elasticity. Preliminary studies on samples from several sources were made, and the nominal values for modulus of elasticity so determined were the basis for selection of deposits from which materials were to be obtained for these tests. The following stones and locations of deposits were chosen:

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[3] 1944 Book of A.S.T.M. Standards, Part II, p. 1.

Material	Source
Bowling Green limestone	Pike County, Mo.
Burlington limestone, low modulus	Carthage, Mo.
Burlington limestone, high modulus	Carthage, Mo.
Plattin limestone	Cape Girardeau, Mo.

The determination of modulus of elasticity on these stones was made by the same methods and apparatus reported by Willis and De Reus (1), which have been described fully in their report and need not be repeated here (Table II).

Large numbers of 1-in. cores on which to make determinations of the modulus were drilled from selected pieces of stone from each quarry. This procedure provided a basis for the careful selection of samples of stone from each source in order to produce an aggregate of uniform physical properties. Considerable variation is shown in samples of stone from a single quarry and hence a large number of pieces had to be examined, and a selection was made of the particular blocks of stone from each source which showed best agreement in the tests for modulus of elasticity (Table II).

TABLE III.—GRADATION OF COARSE AGGREGATES.

Sieve Size, in.	Total Passing, per cent
1½	100
1	88
¾	60
½	27
⅜	13
¼	0

Values of moduli on stones from the several sources are illustrated graphically in Fig. 1. A weighted mean value of modulus for each class of stone was determined.

The values of modulus as determined by tests on the stones selected for aggregates in the concrete mixtures are given in Table II.

The coarse aggregates were then produced from the several stone sources by crushing to a convenient size and screening into several fractions, then recombining to give the gradation shown in Table III.

PROPORTIONS OF CONCRETE MIXTURES

For the purpose of producing accurate proportions of materials, the determinations of amounts of each material, cement, sand, and stone, are based on proportions by absolute volume. Amounts of each material were determined by this method and actual weights were computed for each proportion. This method takes into consideration the specific gravity of each material and weight per unit volume. Greater exactness was thereby obtained in making up each batch of concrete.

TABLE IV.—COMPOSITION OF MIX (BY VOLUME).

Mix	Cement	Water	Fine Aggregate	Coarse Aggregate
I	1	0.77	2.19	3.05
II	1	0.77	2.19	3.57
III	1	0.77	2.19	4.24

In order to bring out as well as possible the effects of the different kinds of aggregates on the properties of the several concrete mixtures, a carefully controlled mortar of uniform character and consistency was used throughout the entire series of samples.

Three mixes were designed for each class of coarse aggregate, varying only the amounts of stone so that the proportions shown in Table IV were obtained. Concrete samples for the tests were molded in cylinder forms, 4 in. in diameter and 8 in. in height.

For comparison with the moduli shown by the concrete samples, mortar samples were also made in 4 by 8-in. cylinders, using the same proportions that were used for the mortar in the concrete containing the various coarse aggregates.

Precautions were taken to insure uniform conditions with regard to effective water in the mixtures. All aggregates were soaked in water for a period of several days to preclude any variation in consistency of mix due to absorption by the aggregates of the water added at the time of making samples of concrete.

The program of tests provided for measuring the differences in modulus of elasticity of the concretes, with relation to the amount of coarse aggregates used in the mixtures made with aggregates derived from a single source. However, the variation in the amounts of coarse aggregates in the three mixes was not very large. The results did not indicate any regular and consistent differences in modulus with regard to the ratios of coarse aggregates, probably explained by the masking effect of the modulus of the mortar matrix in which the coarse aggregates were embedded. Values of modulus determined on individual cylinders made from one class of aggregates in any given mix often showed more variation than that exhibited between any two sets of cylinders in which different proportions of the coarse aggregates were used.

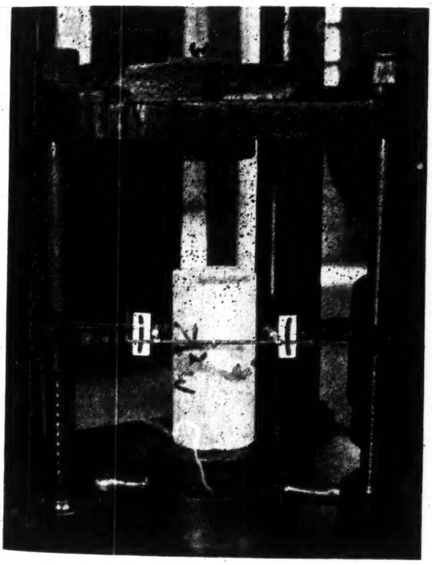

FIG. 2.—Huggenberger Tensometer.

DETERMINATION OF MODULUS OF ELASTICITY

Measurements of the concrete cylinders were made by loading in a universal testing machine. Application of load was started at 500 lb. and increased to 1000, 2000, 3000, etc., up to a maximum of 12,500 lb., which gave a nominal stress of 1000 psi. on the 4-in. cylinders.

TABLE V.—RELATION BETWEEN MODULUS OF ELASTICITY OF CONCRETE AND THAT OF COARSE AGGREGATES.

Type of Aggregate	Modulus of Elasticity of Stone, $E_s \times 10^{-6}$	Modulus of Elasticity of Concrete, $E_c \times 10^{-6}$			Mean Value
		Mix I	Mix II	Mix III	
Limestone Bowling Green........	1.877	3.884	3.924	4.054	3.954
Limestone L.M. Carthage........	6.733	5.094	4.951	5.700	5.208
Limestone H.M. Carthage........	9.047	5.979	5.892	6.650	6.174
Limestone (Plattin)............	11.370	6.913	7.553	6.730	7.065
Sand Mortar.......	$E_m = 5.320$				

Deformation readings were taken at each increment of load with Huggenberger tensometers (Fig. 2). Since these instruments are used in pairs mounted on opposite sides of the cylinders, eccentricity is eliminated in values obtained for deformation.

The scale of the tensometer is graduated to divisions having the value of 0.05 unit on the dial. On this scale, observations to one-fifth division can be easily and accurately estimated for the movement of the needle, thus giving the readings of 0.01 unit. Indicator levers will show movement on the dials for a load as small as 25 lb.

Loadings were carried only to a value well below the elastic limit of the con-

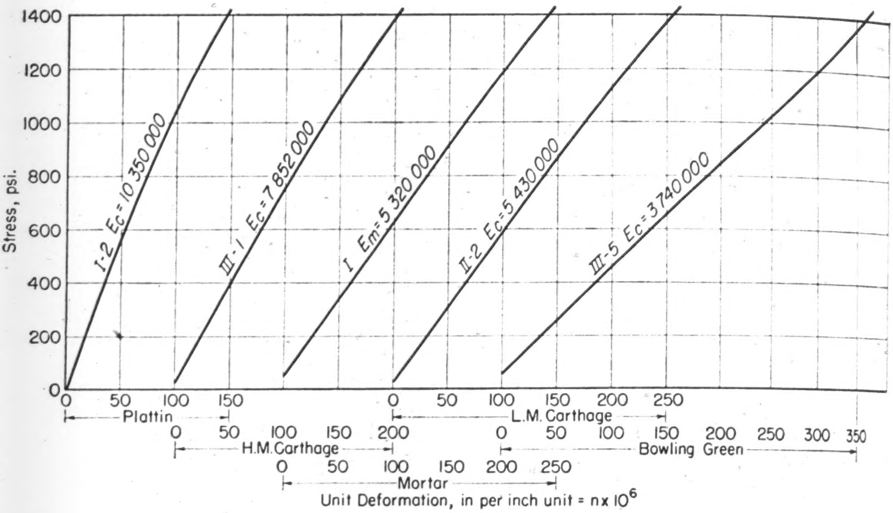

FIG. 3.—Typical Stress-Strain Curves for Concrete Cylinders.

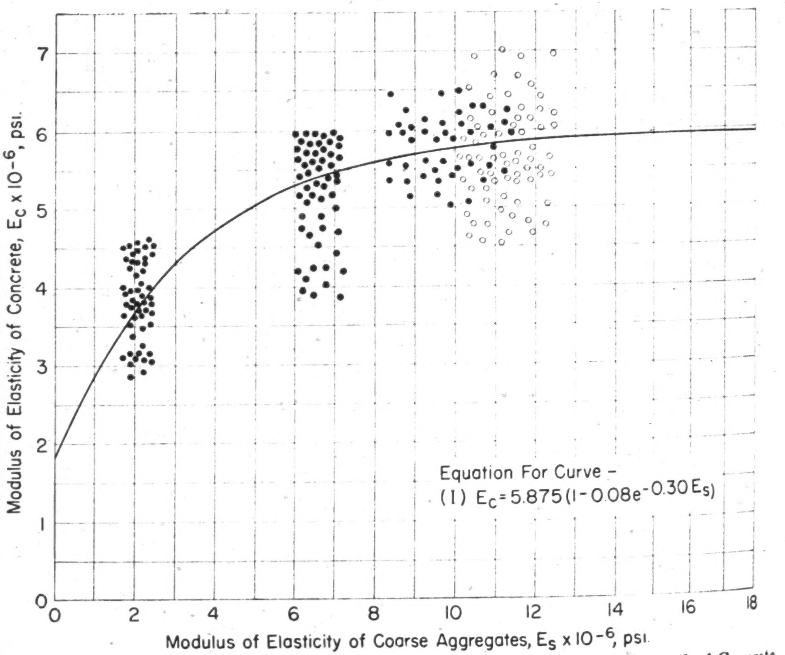

FIG. 4.—Relationship Between Modulus of Elasticity of Coarse Aggregate and of Concrete.

TABLE VI.—COMPRESSIVE STRENGTH IN PSI. FOR 4 BY 8-IN. CONCRETE CYLINDERS.

Plattin Limestone

Age	P I	P II	P III
7 days	852	1178	1127
	942	1054	1062
	1051	1100	1078
28 days	1389	1531	1608
	2068	1562	1390
	1321	1359	1569
	3100	2855	2970
	2660	3490	3250
	2600		3080
	2520		
	2520		
4 yr	4270	6620	5610
	4455	8080	4130
	5690	7460	3690
	5920	9320	4620
	4760	7660	4080
			3890
			4200
			4800
	4995	4240	3480
	4735	4275	2550
	4700	4895	2740
	5490	4000	2460
	5870	3700	3330
	5070	3800	

Burlington Limestone (High-Modulus Carthage)

Age	B I	B II	B III
7 days	1666	1287	953
	1684	1158	940
	1779	1255	1123
28 days	1650	2068	1533
	1940	1561	1424
	1452	1742	1410
	3420	3580	3460
	3090	4045	2980
	3100	3980	3840
	3220	3810	2425
	3620	3620	3390
4 yr	4140	4646	5235
	3830	5621	5840
	4310		5025
	4705		5800
	4210		5880
	3355	3505	2095
	5375	3710	2765
	4070	3110	2955
	4595	3410	
	5550		

Bowling Green Limestone

Age	C I	C II	C III
7 days	1530		
	1530		
	1190		
28 days	3060	2335	1455
	2660	2265	1770
	2670	2405	1915
	2630	1780	1980
	2030	2160	2125

TABLE VI.—*Continued*

Bowling Green Limestone—*Continued*

Age	C I	C II	C III
4 yr	4090	4850	
	3680	4580	
	4615	4570	
	5150		
	5480		5030
	5100		3990
	5000		4670
	4530		4990
	5390		5350
	4490		

Burlington Limestone (Low-Modulus Carthage)

Age	D I	D II	D III
28 days	3105	2940	4045
	3340	2820	2980
	3840	3080	3020
	2985	3440	3300
		3180	3960
4 yr	4650	4610	6220
	6250	5790	4480
	6000	4870	5200
	5910		6670
	4440		6250

(M) Mortar Cylinders

28 days	3460		
	3410		
	3780		
	4050		
4 yr	6700	6800	
	5820	7170	
	6450	7390	
	6950	7630	
	5700	7550	
	6050	7030	
	6350	7750	
	5330		

Note.—Graphic representation of these values is given in Fig. 6. These data do not show any definite relation between the modulus of elasticity and the compressive strength for concrete made from the various aggregates used in this series of tests.

crete, and readings were taken on both ascending and descending loads. In some cases, a lag was shown in deformation readings taken on the descending scale of loads, probably due to a slight degree of plastic flow in the concrete. Nevertheless, the greater number showed very regular and consistent results, with a straight-line ratio of stress to deformation within the limits of loads imposed on the samples. In practically all cases, the values of modulus were computed from the ascending scale of loads and the corresponding deformation readings.

The slopes of the stress-strain graphs plotted for descending readings were parallel to those for ascending scale of values.

Graphs for several of the cylinders are shown in Fig. 3 to illustrate the performances of the concrete under load.

The curve shown in Fig. 4 is plotted from the data obtained from tests of more than 200 concrete cylinders, on which repeated measurements were made totaling over 900 individual tests for modulus of elasticity.

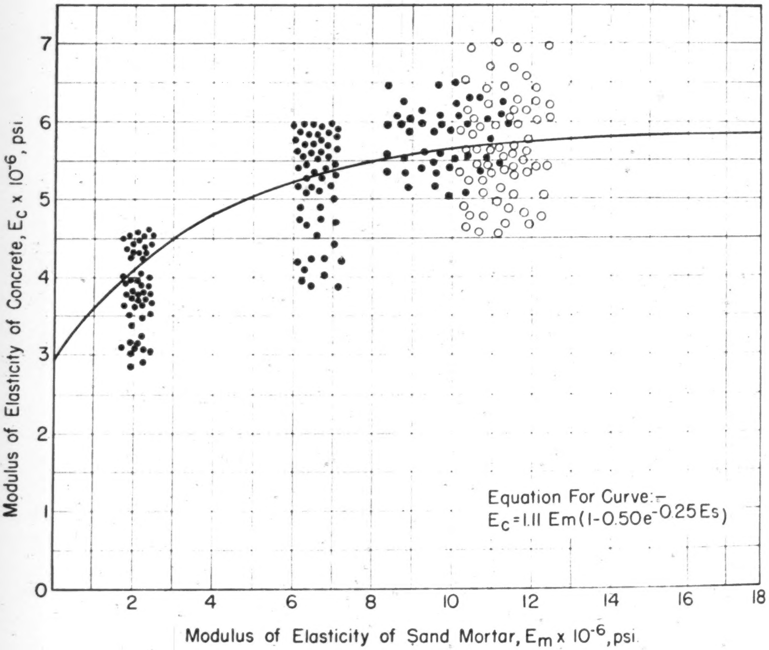

Fig. 5.—Relationship Between Modulus of Elasticity of Sand Mortar and of Concrete.

Equations Derived from Graphs

Two forms of equations may be derived from the curve drawn through the median position of the plotted points; one equation expresses only a relationship between the modulus of elasticity of the stone and that of the concrete in which it is used (Fig. 4); the second equation includes the modulus of the mortar itself as a factor which has slightly different values for the constants from the first equation (Fig. 5). However, the two curves for the expressions do not differ materially in shape and position on the graph sheet.

Compressive Strength of Concrete

Data from the crushing tests on the several mixes in this series (Table VI) do not indicate any consistent relation between the hardness of the aggregates and the compressive strength of the concretes. The concretes in which the softer coarse aggregates were used showed strength equal to those in which hard coarse aggregates were used, in some cases even higher strengths. Because of this evidence, the relations of modulus of elasticity *versus* strength are not included in the development of the theory on characteristics of concrete. Efforts were confined to the determination of the relationships that apparently

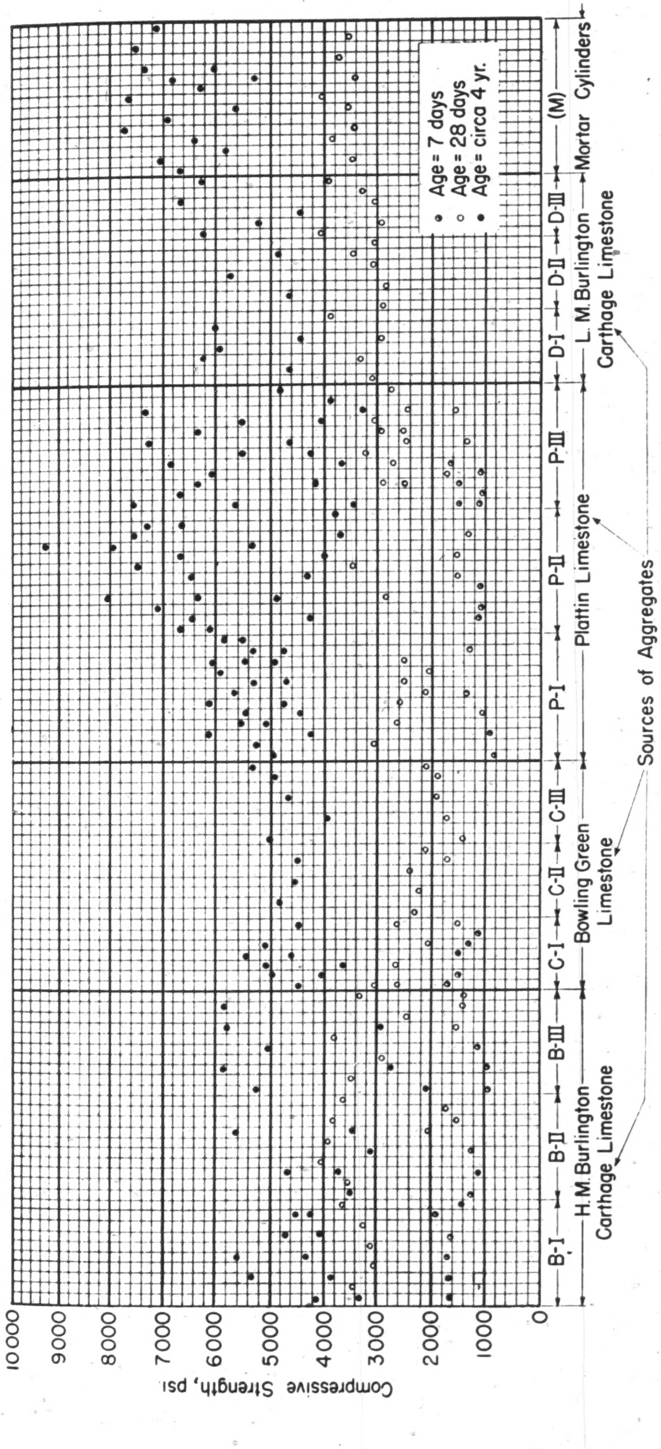

FIG. 6.—Chart of Crushing Tests on 4 by 8-in. Concrete Cylinders.

were shown between the elastic properties of the aggregates, the mortar, and the concrete.

DISCUSSION

Since the preponderance of the data follow the general relation expressed by the equations of exponential type stated previously, they take the general forms:

$$\text{(I)} \qquad E_c = k\,(1 - pe^{-aE_s})$$

and

$$\text{(II)} \qquad E_c = k \cdot E_m\,(1 - pe^{-aE_s})$$

These equations give a fairly accurate mathematical statement of the influence of aggregates on the modulus of the concrete. As the values of E_s increase, the quantity (pe^{-AE_s}) decreases in value, approaching zero as a limit. The constant "k" may also be written (kE_m), which takes account of the modulus of mortar in the concrete. The constant "p" and "a" are dependent on characteristics of the coarse aggregates other than the modulus of elasticity, such as shape of particles, roughness of surface and adherence of mortar to stone particles.

The plotted data show that for increase in values of moduli for the stone, the value E_c (modulus of concrete) increases at a slower rate than that of the stone in the series of mixes in such a manner that E_c approaches a maximum value of approximately 6,000,000 psi. These values no doubt would vary for other aggregates to some extent, also the use of different cement-sand-water ratios in the mortar would be expected to have some influence on the shapes of the curves through the means of the plotted data.

CONCLUSIONS

1. Aggregates produce a decided effect on the elastic properties of concrete, stone having a high modulus of elasticity developing correspondingly higher moduli in the concretes than stone having low moduli.

2. The relation of the modulus of elasticity of aggregates to that of the concrete is not a linear function, but may be expressed by an equation of exponential type, for which the maximum value for the modulus of elasticity of the concrete is approximately 6,000,000 psi.

3. Since the mortar has an important influence on the elastic properties of the concrete, this serves to modify the effects of the aggregates on the moduli of the concretes. The value of modulus for the mortar being approximately median with respect to the range of values on the stones, the concrete made with aggregates of highest modulus had lower modulus than that of the stone, and the modulus of the concrete made with low modulus aggregate was raised above that of this stone.

Fair agreement was shown between the modulus of concrete and the mortar, when stone having approximately the same value of modulus as the mortar was used for aggregates.

4. No correlation between the compressive strength of concrete and the modulus of elasticity is evident from a study of the data.

5. A high degree of precision in measuring deformations is essential to determine differences in moduli for the several concretes, since total deformation for all mixtures is relatively small.

6. Elimination of several variable factors affecting the properties of concrete is necessary in order to evaluate the effects of the elastic properties of the aggregates on those of the concrete. The program of experiments reported herewith was planned to control certain variables not pertinent to this study, with the purpose of eliminating them, or at least reducing their effects to a minimum, thereby accentuating the effects of the modulus of the aggregates

in their influence on the modulus of the concrete.

7. Careful control of water-cement ratio, gradation of aggregates, proportioning of mixtures, mixing, molding, and curing specimens under uniform conditions, makes the moduli of aggregates the primary factor in controlling the moduli of the concrete.

8. Basically, a concrete mixture is a conglomerate, and consequently has many variations in its characteristics, even in different portions of a single mass of concrete. It appears to be impossible, especially in practice, to produce a 100 per cent homogeneous material, even in a single batch of concrete, mixed under the best possible working conditions. Results as shown do not indicate any regular and consistent differences in modulus with regard to the ratios of coarse aggregates in the three different proportions, made from material from any single source. Variation in the amounts of coarse aggregates for the three mixtures was not very great. Values of modulus on individual cylinders of any single mix often varied more widely than that exhibited by any two sets of different proportions.

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DISCUSSION

Mr. N. T. F. Stadtfeld.[1]—How many cylinders were included in one test set—three, six, or nine? How good was the correlation between cylinders in one set?

Mr. H. A. LaRue (*author*).—Three sets of mixtures, thirty to fifty cylinders, in each set. The total number of cylinders on which the data in this report are based was in the order of four hundred or more. We made sets of six and twelve cylinders for each series. Then we repeated operations on certain sets of the series, covering a period of six years, making two or three sets of cylinders for a given mix with one class of aggregates. We molded a total of something like eight hundred to one thousand test cylinders, on which measurements for deformations under load were repeated several times. A large number of these tests I made with my own hands and can vouch for the accuracy of the results.

I have observed in our testing work, that variations in concrete are unlimited. Covering the past twenty or thirty years, our records include reports on construction projects from all over the state. Some of the engineers on the state jobs had to regulate their concrete production very carefully, conforming to specifications set up by the W.P.A. and other governmental agencies. There were all sorts of variations in strengths, where they were supposed to be controlling the production of concrete under very rigid specifications.

Recently, on extensive improvements in Columbia, Missouri, concrete foundations for a million-gallon water tower were constructed. The engineer on this job has been all over the country on different projects, and has had broad experience on concrete work. He set up rigid control on this work, checking the scales at the central mixing plant with our 50-lb. standard weights, even going to the extent of having his plant inspector operate the levers on the scales for batching the concrete.

Preliminary trial mixtures were selected and tested in our laboratory, to determine proportions of the concrete. Consistently high strengths were obtained on the construction. Some of these strengths ran as high as 6000 psi.

Mr. Stadtfeld.—What was the percentage of variation between say twelve cylinders?

Mr. LaRue.—Sometimes 50 per cent plus or minus, for the extreme range of values. However, many cylinders were discarded which showed faults and seams due to conditions of molding. The values of modulus for the concrete samples used in this report were selected from the more consistent values obtained on sound cylinders. Variation from the median value on a selected set of cylinders was 20 per cent, or less.

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[1] Division Engineer, Board of Water Supply, New York, N. Y.

THE USE OF THE DYNAMIC MODULUS OF ELASTICITY IN PREDICTING THE 28-DAY FLEXURAL STRENGTH OF CONCRETE*[1]

By Edmund F. Preece[2]

Synopsis

For a given set of conditions there is a relationship between the dynamic modulus of elasticity[3] and the age of a concrete test specimen. In addition, the dynamic modulus and the coincident flexural strength are related also. Together, these relationships provide a means for predicting the 28-day flexural strength of a concrete test specimen when it is but a few days old.

In the Corps of Engineers' procedure for the design of rigid airfield pavements (**1**),[4] the slab thickness is a function of the required load capacity, the modulus of soil reaction of the base course, and the flexural strength of the concrete. At the time the concrete is poured, both the load requirements and the soil reaction are known, but an assumed value must be used for flexural strength. Where, for one reason or another, that assumed value provides but a small factor of safety, it is desirable to check the accuracy of that assumption as early as possible.

The estimating procedure herein described was developed in connection with a job for which the assumed flexural strength value was questionable at best. It was job-developed and is wholly empirical in nature. It is being presented simply as a workable job-control measure. This is emphasized to explain why the data presented are rather limited and also why certain inferences to be drawn from the data presented have not been explored in greater detail.

Test Specimens

Normally three test beams are prepared to represent each day's pour or each 1000 sq. yd. of pavement if the daily production exceeds this amount. The procedure described here was anticipated before pouring started and during the first few pouring days six beams were prepared daily. Of these, three were reserved for 28-day breaks and the balance of each day's beams were used for earlier breaks.

The nominal dimensions of the test beams were 6 by 6 by 30 in. and 6 by 6 by 36 in. The number of beams cast required the use of every available form and no other significance attaches to the difference in length.

All beams remained on the job and in the forms until the second day. The purpose of this delay was to reduce the possibility of damage through too early handling. As soon as they were stripped, the beams were protected from drying in transit and removed to the laboratory where they were stored in the humid room until they were broken. The importance of careful handling and proper curing will be discussed later.

Test Procedure

Both the apparatus and the computational procedure used in the determina-

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] The use of this paper for publication has been approved by the War Department.

[2] Chief, Branch of Civil Engineering, U. S. Engineer Office, Corps of Engineers, U. S. Army, Washington, D. C.

[3] Hereinafter referred to as the "dynamic modulus" for convenience.

[4] The boldface numbers in parentheses refer to the references appended to this paper, see p. 1319.

tion of the dynamic modulus have been described comprehensively elsewhere (**3, 4, 5, 6, 7**). For the tests reported here, an assembled apparatus similar to that described by Willis and De Reus (**4**) was used. This apparatus consisted essentially of a Beat-Frequency audio oscillator, a Fixed-Frequency audio oscillator, a 15-w. power amplifier, a Class A amplifier, a 3-in. oscillograph, and a 6-w. Jensen permanent magnet loud speaker.

TABLE I.—DYNAMIC MODULUS OF ELASTICITY VALUES.

Axis *d* = Depth As Cast
Axis *b* = Width As Cast

Sample	Age, days	Dynamic Modulus of Elasticity, psi.		
		Axis *d*	Axis *b*	Average
No. G5/258.....	11	4 530 000	4 730 000	4 630 000
	14	4 530 000	4 850 000	4 690 000
	21	5 020 000	5 020 000	5 020 000
	28	5 020 000	5 080 000	5 050 000
No. G5/281.....	9	3 840 000	4 060 000	3 950 000
	14	4 370 000	4 320 000	4 340 000
	21	4 340 000	4 600 000	4 470 000
	28	4 470 000	4 730 000	4 600 000
No. G5/282.....	9	3 940 000	3 760 000	3 850 000
	14	4 400 000	3 990 000	4 200 000
	21	4 540 000	4 180 000	4 360 000
	28	4 400 000	4 380 000	4 390 000
No. G5/284.....	9	3 790 000	3 930 000	3 860 000
	14	4 070 000	4 350 000	4 210 000
	21	4 260 000	4 500 000	4 380 000
	28	4 460 000	4 700 000	4 580 000
No. G5/294.....	7	3 820 000	3 760 000	3 790 000
	14	4 350 000	4 660 000	4 500 000
	28	4 840 000	4 840 000	4 840 000

The specimen holder, however, was constructed throughout of oak in order to insure a low natural frequency well below that to be expected for the large beams.

For each reported value of the dynamic modulus, the resonant frequency was determined for two axes normal to each other. For computational purposes the cross-sectional dimensions were determined to the nearest 0.02 in., the length to 0.05 in., and the weight to 0.25 lb. The dynamic modulus with respect to each of the normal axes was determined individually using the appropriate dimensions. The recorded value was the average of the two individual values. Typical data are shown in Table I.

Dynamic Modulus - Age Relationship

The use of the dynamic modulus to indicate the decay of strength in the freezing-and-thawing test has been reported by a number of investigators (**3, 4, 8, 9, 11, 14**). A large number of 3 by 4 by 16-in. beams tested in connection with an earlier project had shown that it was equally suitable to indicate the increase in strength during the curing period.

Typical data are shown in Fig. 1. The curves of group I represent the concrete used for the subject work; group II represents a concrete in which a better type of coarse aggregate was used but which is similar to that represented by group I in all other respects. Normal inspection controls were exercised and the variations in water-cement ratio, cement content, and batch weights were neither greater nor less than that to be expected on any well-controlled work. The principal variable, therefore, was the quality of the coarse aggregate and the effect of this factor is emphasized by the relation of groups I and II.

It may be noted in Fig. 1 that the curves are approximately parallel after 14 days. The average slope is indicated by the slope of *A-A′* and *B-B′* which have been added to mark the upper and lower limits of group I respectively.

In Fig. 2, data presented by Axon, Willis, and Reagel (**10**) have been plotted in the same manner as the data of Fig. 1. These curves represent concrete of three different air contents for each of four different coarse aggregates, two of which were chert and the others limestone. The enveloping curves of Fig. 1, *A-A′* and *B-B′*, have been added to permit direct comparison of the data.

Data which have been presented by

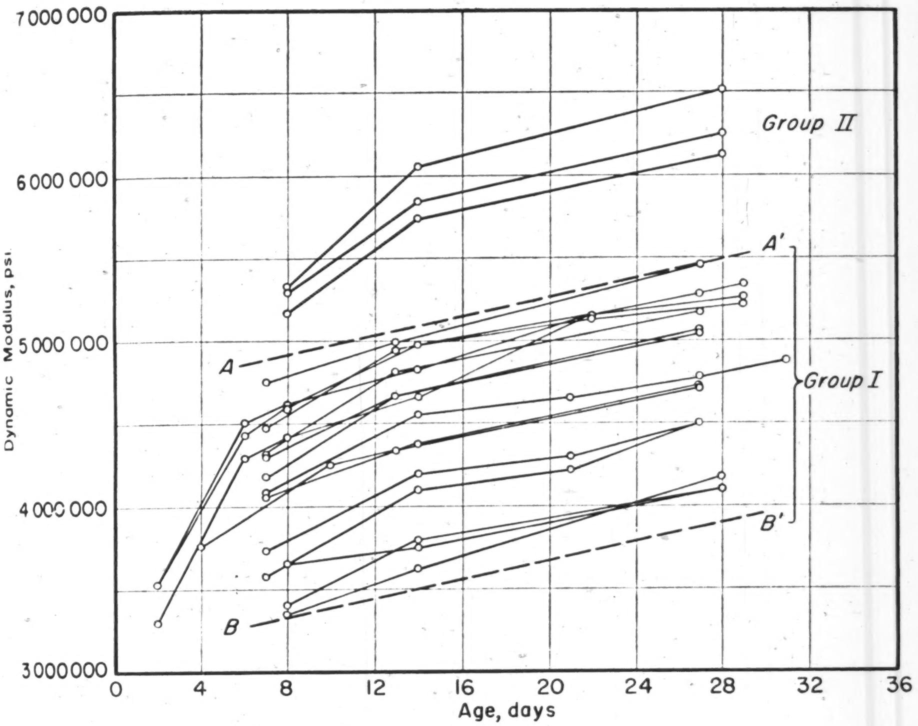

FIG. 1.—Dynamic Modulus-Age Relationship.

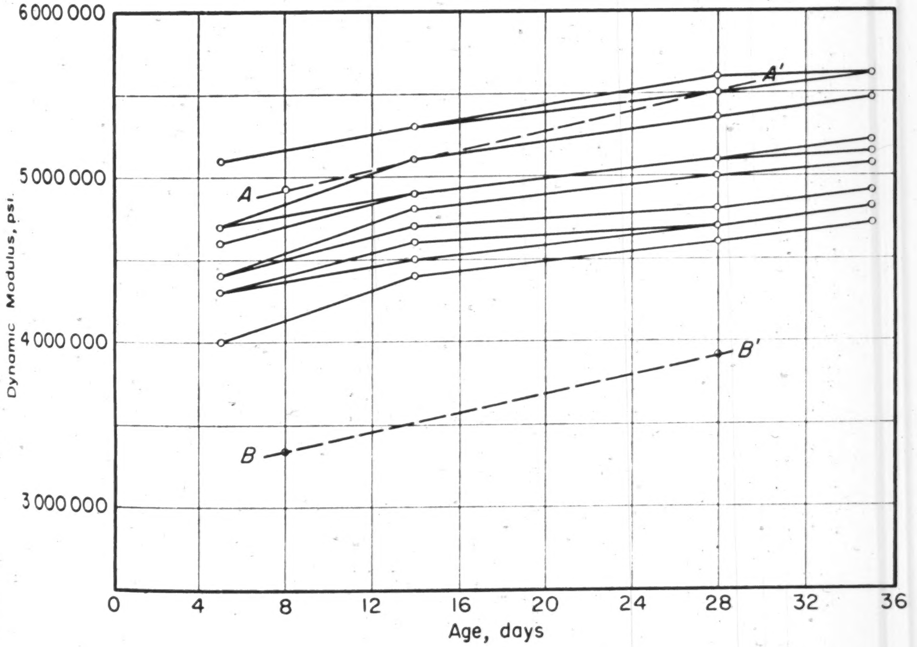

FIG. 2.—Dynamic Modulus-Age Relationship. (After Axon, Willis, and Reagel (10).)

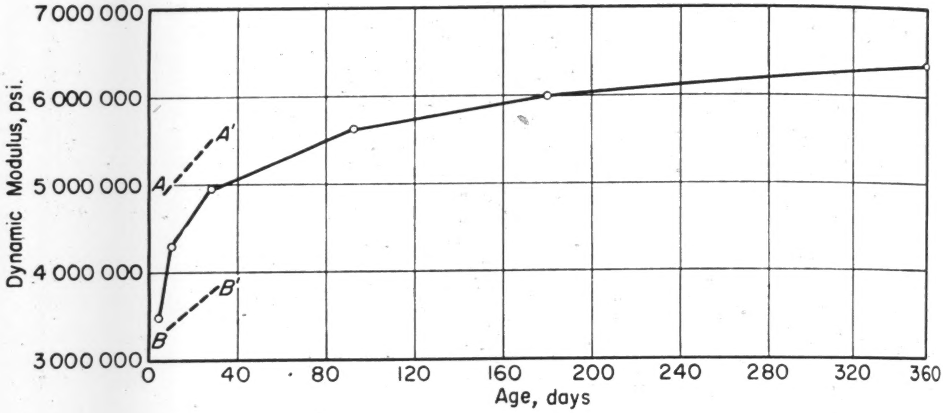

FIG. 3.—Dynamic Modulus-Age Relationship. (After Stanton (**13**).)

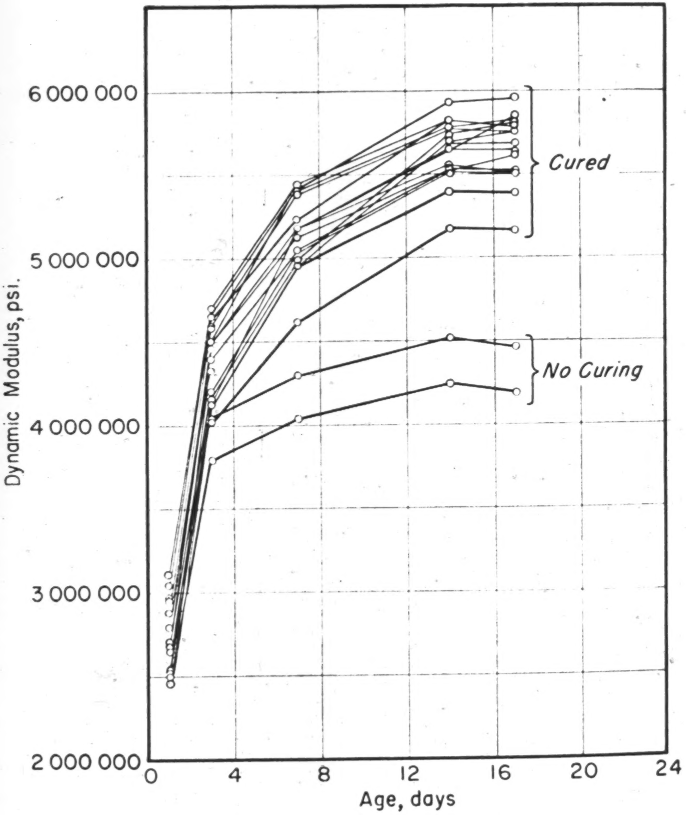

FIG. 4.—Effect of No Curing. (After Long and Kurtz (**14**).)

Stanton (**13**) are shown in Fig. 3. Each of the plotted points indicated by small circles represents the average for three specimens and for the corresponding period the curve is similar to those of Figs. 1 and 2.

The data presently available appear to furnish substantial reason to believe that the average development of the strength of concrete with age as indicated by the dynamic modulus is generally uniform for a given set of conditions. Sufficient data are not now available definitely to define the permissible tolerance in each of the factors which go to make up a given set of conditions, but it is probable that the individual limits are restrictive.

Munger (**12**), Kellerman (**15**), and Walker (**16**) among others have discussed the effect on concrete of variations in the quality of the coarse aggregate. In all probability, different types of aggregate will produce different families of curves similar to those of Figs. 1 and 2, but variations in the quality of a given type, other things being equal, will probably be reflected only in the spread between the minimum and maximum values. Cement content, water-cement ratio, and batch proportions may have a greater or a lesser effect. But it seems likely, however, that the range of variation in these factors to be expected with ordinary control measures

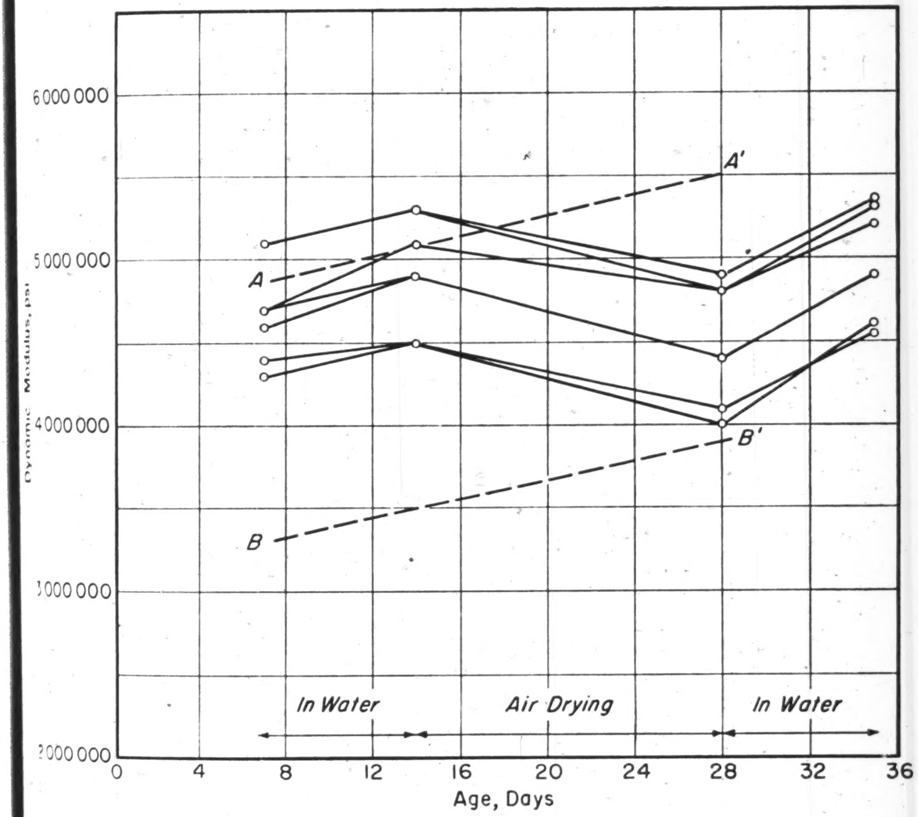

FIG. 5.—Effect of Air Drying. (After Axon, Willis, and Reagel (**10**).)

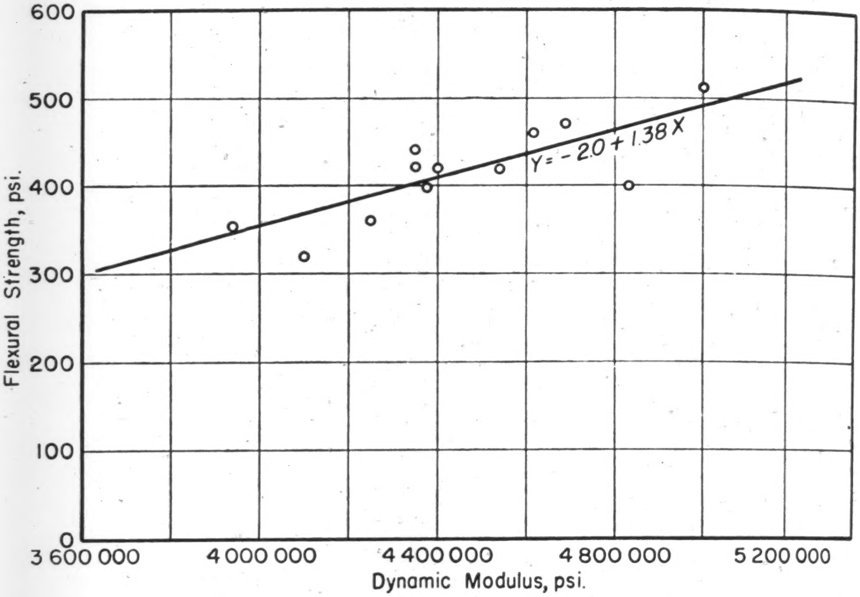

FIG. 6.—Dynamic Modulus-Flexural Strength Relationship.

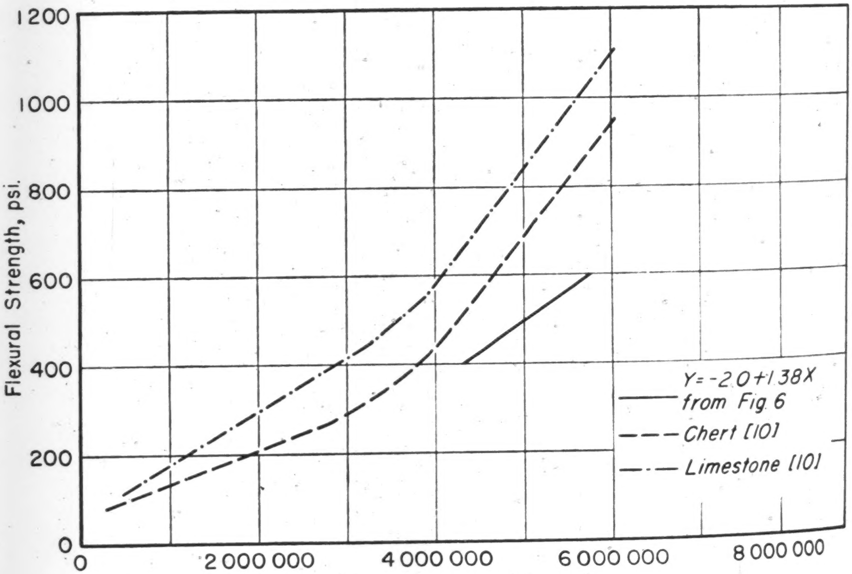

FIG. 7.—Dynamic Modulus-Flexural Strength Relationship. (After Axon, Willis, and Reagel (10).

does not influence the relationships significantly.

There is one important factor, however, to which special attention is called. Data presented by Long and Kurtz (14) are shown in Fig. 4, and Fig. 5 has been prepared from data presented by Axon, Willis, and Reagel (**10**). Both figures emphasize the importance of proper curing to the dynamic modulus - age relationship. It is readily apparent in these data that drying of the specimens must be avoided if a consistent relationship among the age, the dynamic modulus, and the flexural strength is the purpose of the tests.

TABLE II.—DYNAMIC MODULUS OF ELASTICITY AND FLEXURAL STRENGTH FOR EARLY-BREAK SPECIMENS.

Sample	Breaking Age, days	Dynamic Modulus of Elasticity, psi.	Flexural Strength, psi.
No. G5/220	17	4 610 000	460
No. G5/233	16	3 940 000	350
No. G5/237	15	4 250 000	360
No. G5/253	14	4 690 000	470
No. G5/254	14	5 000 000	510
No. G5/280	14	4 830 000	400
No. G5/283	14	4 400 000	420
No. G5/285	21	4 380 000	400
No. G5/293	14	4 350 000	440
No. G5/303	16	4 350 000	420
No. G5/332	14	4 540 000	420
No. G5/344	14	4 100 000	320

DYNAMIC MODULUS - FLEXURAL STRENGTH RELATIONSHIP

Since both the flexural strength and the dynamic modulus reflect the same qualities of the specimen, it is to be expected that there is a relationship between them. Such a relationship was shown by Willis and De Reus (**4**) expressed in terms of the loss of strength. Stanton (**13**) gives additional evidence, and Munger (**12**) found a good correlation but concluded that it was not a straight-line function.

It was mentioned earlier in discussing the test specimens that more beams were cast during the first few days of pouring than were needed for 28-day tests. Most of these extra beams were broken at about 14 days in order to obtain pairs of dynamic modulus and flexural strength values of a considerably lower order of magnitude than was expected at 28 days. The purpose of this was to obtain as wide a spread in the pairs of values as was practicable in order better to define the relationship. The early-break data are tabulated in Table II.

The relationship is not directly apparent in the tabulation, although it can be determined readily by the method of least squares. Plotted, however, as in Fig. 6, the relationship can be seen as soon as sufficient points are available to define it and, in addition, it permits of visual determination of the average curve.

Axon, Willis, and Reagel (**10**) determined the dynamic modulus - flexural strength relationship for concretes having limestone and chert aggregates. Their graphs together with the average curve of Fig. 6 are given in Fig. 7. It is to be noted that the relationships are not linear functions, as Munger (**12**) also found. Nevertheless, for flexural strength values greater than about 450 psi. the relationship by Axon, Willis, and Reagel (**10**) is essentially linear, and even between 300 and 450 psi. a linear relation could be assumed without too great an error for a limited range between the maximum and minimum values. Since a linear relationship can be assumed over a relatively short range of values in any case, it follows that a plotting such as Fig. 6 is practical even if not extended to 0, 0.

The factors which affect the dynamic modulus - age relationship also affect the dynamic modulus - flexural strength relationship, although not in the same degree. Data are available (but in too fragmentary a form for presentation at this time) which agree with the relationship found by Axon, Willis, and Reagel (10), that is, that coarse aggregates of different structural characteristics will have a unique dynamic modulus - flexural strength relationship in the same manner that there are two distinct relationships in Fig. 7, one for the chert and the other for the limestone. If the data obtained in the tests under discussion had been as complete, it is probable that a third curve would have been defined comparable to but distinct from the others throughout its length.

The steps taken to reduce the possibility of damage to the test beams through too early handling was pointed out earlier. A crack in a specimen may have a very important effect upon the flexural strength but, as pointed out by Kirmser (2), the effect on the natural frequency of the specimen and consequently its dynamic modulus may be negligible—particularly if the crack is very narrow or is filled with dirt or crystallized material. Obviously, anything which affected the two qualities unevenly would destroy the relationships being sought.

Drying during the curing period would probably have the same effect as cracks, that is, the points in the dynamic modulus - flexural strength relationship would be so scattered that they would serve only to confuse.

The Estimating Procedure:

Once the slope of the dynamic modulus - age pattern and the dynamic modulus - flexural strength average line are determined for a particular set of conditions, the estimating procedure is very simple. An example will best illustrate the procedure:

Assume that group A of Fig. 1 and the curve of Fig. 6 represent the particular concrete being tested. Assume further that the dynamic modulus of a specimen 14 days old has been determined to be 4.50×10^6. If from this point in Fig. 1 a line is drawn parallel to the average slope *A-A′* or *B-B′* it will be found to intersect the 28-day ordinate at a probable value of 5.07×10^6. Next, entering Fig. 6 with this probable value for the dynamic modulus, the corresponding estimated value of the flexural strength at 28 days is found to be 500 psi.

CONCLUSION

From this investigation the following conclusions are made:

The resonant frequency test, which has been used principally to reflect the decay of strength of concrete, can be used to disclose the development of strength.

For a given aggregate, batch proportions, type of cement, and water-cement ratio, the relationship between the development of the dynamic modulus of elasticity and the age in days appears to be consistent for practically all specimens. Of the various factors which may influence the relationship, variations in the character of the coarse aggregate appear to be the most important.

For a given set of conditions, the relationship between the dynamic modulus of elasticity and the flexural strength is a linear function, with this relationship apparently being different for different coarse aggregates and probably for different cements, proportions, and slump also.

Proper curing, including the avoidance of drying of the test beams, and care in their early handling to prevent cracks, is very essential in avoiding a distortion of both the dynamic modulus of elasticity - age and the dynamic modulus of elasticity - flexural strength relationship.

Once the two relationships are very definitely established for a given set of local conditions by appropriate tests on 6 by 6 by 30-in. beams, further tests for stimating the 28-day flexural modulus may be made on small 3 by 4 by 16-in. specimens with all the advantages that the use of the smaller specimens implies. The procedure which has been described was devised to serve as a job control and as such it served a definitely useful purpose. It is believed that that usefulness can be increased greatly by further application under a wider variety of conditions than is afforded by a single locality.

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The author desires in particular to express his deep appreciation to the authors to whom reference has been made and whose data have been quoted.

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A STUDY OF DURABILITY AND VOID CHARACTERISTICS OF CONCRETES CONTAINING ADMIXTURES, PRINCIPALLY OF THE AIR-ENTRAINING TYPE*

BY F. B. HORNIBROOK,[1] HOWARD FREIBERGER,[2] AND ALBERT LITVIN[3]

SYNOPSIS

Six different air-entraining admixtures were added in turn to small successive portions of two different cement clinkers which were then ground in a laboratory ball mill. Two additional admixtures were added to the ground plain cement at the concrete mixer. The cements were made into two series of concretes; one series a nominal $5\frac{1}{2}$ bag per cu. yd. and the other a nominal 7 bag per cu. yd. concrete. The air content, slump, flow, and specific weight of the plastic concrete were measured. The resistance to cycles of freezing and thawing, the voids as determined by the air-expansion volumenometer, the saturation coefficients, the permeability to air, and the flexural strengths were determined on specimens of matured concretes. The air contents of the concretes varied over a wide range as did the cement factors, the flexural strengths, the durability factors, and the saturation coefficients. The air permeabilities as measured were of the same order for all the concretes.

In 1938 an investigation was started at the National Bureau of Standards in which a number of admixtures, principally of the air-entraining type, were interground with portland-cement clinker or were added to the finished cement. Properties of concretes made with these cements were then studied. The investigation was not completed or reported because of interruption by prewar defense activities and then by the war itself. Although when the investigation was started the beneficial effects of air-entrainment on workability and durability of concrete were becoming recognized, the currently accepted concepts (**1**, **2**)[4] concerning the optimum amount of entrained air, and the need for re-design of the mix to maintain the cement content per cu. yd. of concrete (cement factor) constant, were not generally recognized. Accordingly, some of the test procedures lacked refinements that would be standard practice at the present time. Nevertheless, some of the data, such as that on air permeability, and on pore space determined by the air-expansion volumenometer, are of a type not heretofore available for concrete. The data on the amount of air entrained by the various admixtures and the effects on strengths and rability factors also may be of special terest. Accordingly, such results as ere completed are presented.

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[1] Master Builders Co., Cleveland, Ohio; formerly with the National Bureau of Standards, Washington, D. C.

[2] U. S. Navy; formerly with the National Bureau of Standards, Washington, D. C.

[3] National Bureau of Standards, Washington, D. C.

[4] The boldface numbers in parentheses refer to the references appended to this paper, see p. 1332.

MATERIALS

ements:

The tests covered in this report were ade on cements prepared in the labora- ry from two commercial clinkers ob- ined from different sources. The emical compositions after the addition gypsum and grinding were as follows:

	Cement No. 2	Cement No. 3
SiO_2, per cent	21.6	19.3
Al_2O_3, per cent	5.5	6.3
CaO, per cent	65.0	64.2
MgO, per cent	1.3	3.7
Fe_2O_3, per cent	2.6	2.4
SO_3, per cent	1.6	2.1
Na_2O, per cent	0.05	0.24
K_2O, per cent	0.12	0.66
Free lime, per cent	0.6	1.36
Loss on ignition, per cent	1.3	1.1
Insoluble matter, per cent	0.1	0.1

dmixtures:

The following materials (in addition gypsum) were interground with suc- essive portions of clinker:

Designation	Material
	Proprietary resin
	Calcium stearate from fish-oil fatty acids
	Ammonium stearate
	Tallow
	Sulfonated tallow
	Waste sulfite liquor

The following two materials were added at the mixer:

	Technical sodium laurel sulfate
	Hydroxylated organic salt

Excepting materials D and L, all of the above admixtures caused appreciable air-entrainment.

ggregates:

A local sand (known as Massaponax sand, fineness modulus 2.7), and local Potomac River gravel, were used. The tter was combined to consist of 33 per ent of the No. 4 to $\frac{3}{8}$-in. fraction and per cent of the $\frac{3}{8}$ to $\frac{3}{4}$-in. fraction. All aggregates were dried before using.

PROCEDURE AND TEST METHODS

Preparation of Cements:

Separate batches of the two clinkers, ne for each of six different admixtures, were ground in a laboratory ball mill to a surface area of approximately 1800 sq. cm. per g. as determined with a Wagner turbidimeter. Three batches of each clinker were also ground without admixture, one batch to be used as the reference cement and two batches for use with admixtures added at the mixer.

Concrete Mixes:

Two series of concretes, designated as the X concretes and the Y concretes, differing in proportions of cement and aggregates, were made for each of the cement-admixture combinations. The X concretes were proportioned 1:2.4:3.6 parts by weight of cement, sand, and gravel. In the plain concrete (without admixture) these proportions yielded a nominal cement factor of approximately $5\frac{1}{2}$ bags per cu. yd.

The Y concretes were proportioned 1:1.75:2.75 parts by weight of cement, sand, and gravel. This mix yielded a nominal cement factor for the concrete, without admixture, of 7 sacks per cu. yd.

No attempt was made to redesign the concretes containing the air-entraining admixtures to maintain the cement factors constant. An attempt was made, however, to adjust the mixing water to hold the slump between 3 and 4 in. Because of the unexpected behavior of some of the concretes, their slumps fell outside the above limits. Since the supply of cement containing each admixture was limited, no duplicate batches could be made.

Mixing, Testing, and Placing of Plastic Concrete:

The concrete was mixed for 5 min. in an open-bowl Lancaster mixer. The slump and flow, and the air content by the method described by Pigman (**3**), were determined. This method uses a vacuum to replace the air with water.

The specific weight was determined as a part of the air-content test. Upon completion of the slump and flow measurements the concrete was remixed and twelve brick-shaped specimens $2\frac{1}{16}$ by $3\frac{3}{4}$ by $8\frac{1}{16}$ in., and two bars, 3 by $3\frac{1}{2}$ by 12 in., were cast. The purpose of preparing specimens of brick shape was to utilize available equipment for the determination of air permeability, and of pore space by the air-expansion method (**4**).

Curing and Testing Program:

All specimens were removed from the molds after one day and stored in the fog room until 14 days old. Three brick-type specimens and one bar were then immersed in water for one day and then subjected to cycles of freezing and thawing. Two brick-type specimens were placed in the laboratory to air-dry before being tested in the air permeameter and the volumenometer and for water absorption. Two brick-type specimens and one bar were kept in the fog room for 34 days (age 35 days) when they were broken in flexure by center-point loading. The remainder of the specimens were left in the fog room for possible future use.

Description of Freezing-and-Thawing Tests:

After the one-day soaking period (age 15 days) the resonant frequency of each specimen was determined for calculation of its dynamic modulus of elasticity (designated as dynamic *E*). The specimens were then inclosed in snug-fitting rubber containers, a measured amount (approximately 400 ml.) of water added to each, and the containers were then placed under water to remove the air, and sealed.

The freezing-and-thawing cycle consisted of 2 hr. of immersion in a circulating alcohol-water mixture maintained at −20 F., followed by 2-hr. immersion in a water tank at 70 F. Two cycles were completed per working day, with the specimens remaining in the thawing bath overnight and over week ends. At selected intervals, depending on the rate of deterioration of the specimen, the resonant frequencies were redetermined. The test was terminated for each specimen either when the dynamic *E* had decreased more than 25 per cent (designated as failure), or at 160 cycles if failure had not occurred. At the end of the freezing test, three of the four test specimens were broken in flexure by center-point loading. The remaining specimen was set aside to dry for subsequent tests of pore characteristics.

The resistance to cycles of freezing and thawing was evaluated by two types of durability factors, a dynamic modulus durability factor, *DFE*; and a strength durability factor, *DFS*. The dynamic modulus durability factor *DFE* was obtained by first plotting the change in dynamic modulus with cycles of freezing and thawing; the ratio times 100 of the area under the curve to the total area of the rectangle formed by the ordinate representing 25 per cent drop in dynamic *E* and the abscissa representing 150 cycles of freezing and thawing was taken as the durability factor. This is essentially the method of calculating the durability factor described by Stanton Walker (**5**) except that the limiting values of drop in dynamic modulus and in the number of cycles were changed in this case to 25 per cent and 150 cycles, respectively.

The strength durability factor *DFS* was calculated from the following formula:

$$DFS = \frac{M_2}{M_1} \times \frac{N_2}{160} \times 100$$

where:

M_1 = modulus of rupture of reference concrete at 35 days (the AX specimen for the X series and the AY specimens for the Y series),

M_2 = modulus of rupture of the representive concretes after completion of freezing-and-thawing test, and

N_2 = number of cycles of freezing and thawing.

By referring the modulus of rupture values after freezing and thawing to the 35-day value obtained for the reference concrete, rather than the 35-day value of the particular concrete under consideration, some degree of evaluation is made of the sacrifice in strength accompanying air entrainment.

Determination of Pore Space:

Solid volumes were determined by means of an air-expansion volumenometer. The tests on specimens made with cement No. 2 utilized the volumenometer described by Stull and Johnson (4). The tests on specimens made with cement No. 3 were conducted in a constant-temperature room using an improved volumenometer described by Richmond and co-workers (6). The determination consists essentially of placing the specimen in a system of known volume at atmospheric pressure. The system is then closed, expanded by a known volume and the resulting change in pressure measured. Since the product of pressure times volume remains constant, the solid volume of the specimen can be calculated.

It is highly essential in this type of measurement that the specimen be at moisture equilibrium with the ambient atmosphere. If the specimen has been oven dried immediately prior to the test, it will adsorb moisture during the test (unless arrangements are made to use desiccated air), and consequently the results will be affected. Similarly if the specimen contains excess moisture it will lose sufficient moisture during the test to affect the results. In this investigation, the specimens were allowed to air dry 6 weeks or longer before test.

Ordinarily about 10 min. were required for the air within the specimen to reach apparent equilibrium with the expanded air in the test system. Some of the more dense specimens, however, required as long as 20 min. to $\frac{1}{2}$ hr. It is desirable, therefore, that the test on concrete specimens be conducted in constant-temperature surroundings.

The bulk volume was measured by determining the weight required to just hold the specimen submerged in mercury, the sum of the specimen weight plus the applied weight, divided by the density of the mercury giving the bulk volume of the specimen. The pore space was then found by subtracting the solid volume from the bulk volume.

Measurement of Air Permeability:

The apparatus used in determining the air permeability was also described by Stull and Johnson (4). It consists essentially of a device for applying a selected constant air pressure to one face of the specimen. The specimen was placed on a spacing gasket to keep the bottom face open to the applied air pressure and then the sides of the specimen were surrounded with mercury up to the top face to prevent escape of air through the sides. The amount of air passing through the specimen was calculated from the pressure difference across the specimens and that across a calibrated capillary tube in series with the specimens. The tests were made on specimens air dried for 6 weeks or longer.

The permeability, f, is expressed as the rate of flow through a 1-cm. cube with unit pressure difference across the cube. It is defined by the equation:

TABLE I.—PROPERTIES OF PLASTIC AND HARDENED CONCRETES AS AFFECTED BY VARIOUS ADMIXTURES.

Designation	Admixture	Plastic Concrete						Hardened Concrete											
		Net Water-Cement Ratio, w/c, by weight	Slump, in.	Flow, per cent	Specific Weight, lb. per cu. ft.	Measured Air Content, per cent by volume^a	Cement Factor, bags per cu. yd.	Porosity, average of two specimens, per cent^b		Saturation Coefficient, average of two specimens		Number of Cycles of Freezing and Thawing, average of four specimens	Drop in Dynamic *E* at End of Freezing Test, average of four specimens, per cent	Modulus of Rupture at End of Freezing Test, average of three specimens, psi.	Modulus of Rupture, Damp-Cured Specimen, 35 days, average of three specimens, psi.	Durability Factors^g		Air Permeability^b	
								Air Expansion Method^c	Water Absorption Method^d	Water Absorption Method^e	Absorption-Air Expansion Method^f					*DFE* Based on Drop in Dynamic *E*	*DFS* Based on Drop in Modulus of Rupture	Reference Specimens, average of two, ml. per sec. × 10^{-5}	One Specimen After Freezing Test, ml. per sec. × 10^{-5}
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
								Cement No. 2, Nominal 5½-Bag Mix											
X-2-A	None	0.55	3	56	145.5	2.3	5.5	12.6	12.4	87	85	28	29	340	670	12	9	5.5	9
X-2-B1	0.02 per cent proprietary resin	0.53	3	48	141.9	5.4	5.4	14.7	13.3	78	71	161	3	520	635	92	78	2.0	79
X-2-B2	0.04 per cent proprietary resin	0.51	4¾	56	134.2	10.2	5.1	19.2	16.5	57	49	160	15	355	505	78	53	4.5	...
X-2-B4	0.05 per cent proprietary resin	0.53	3½	58	138.2	10.8	5.3	16.2	13.0	76	62	160	10	410	610	87	61	2.2	...
X-2-C	0.04 per cent fish-oil stearate	0.55	6	70	137.4	7.8	5.2	17.6	15.9	66	60	160	6	425	580	96	64	4.4	...
X-2-D	0.07 per cent amm. stearate	0.54	2½	42	147.5	1.8	5.6	11.0	11.5	92	96	32	28	345	770	13	10	1.5	43
X-2-E	0.035 per cent tallow	0.55	4¼	59	142.5	4.5	5.4	14.5	14.2	76	75	106	32	275	580	42	27	3.9	...
X-2-F	0.05 per cent sulfonated tallow	0.55	3	58	145.0	3.4	5.5	12.6	13.3	85	91	128	25	330	590	62	39	5.5	...
X-2-J	0.02 per cent waste sulfite liquor	0.51	3½	54	139.5	6.2	5.3	14.2	11.8	86	71	161	18	460	600	79	69	4.1	...
X-2-K	0.025 per cent sod. laurel sulfate	0.51	3¼	51	140.1	6.5	5.4	16.2	14.6	68	61	160	16	370	585	91	55	4.1	...
X-2-L	0.1 per cent hydroxylated organic salt	0.54	3	52	146.2	2.3	5.5	12.0	12.3	89	91	36	26	330	630	12	11	1.8	8
								Cement No. 3, Nominal 5½-Bag Mix											
X-3-A	None	0.55	2½	57	146.8	1.6	5.6	12.5	10.1	90	73	13	32	325	730	4	4	2.3^h	25
X-3-B1	0.02 per cent proprietary resin	0.51	5	61	138.0	7.8	5.2	15.6	11.7	76	59	161	12	405	565	91	55	2.3	95
X-3-B2	0.04 per cent proprietary resin	0.49	4¾	53	127.2	10.2	4.9	24.7	22.2	38	34	160	11	310	370	83	42	22.	91
X-3-C	0.035 per cent fish-oil stearate	0.50	4½	59	134.2	9.7	5.1	20.0	14.6	60	44	160	7	390	510	93	53	3.3	47
X-3-D	0.1 per cent amm. stearate	0.52	2½	51	145.5	3.2	5.4	11.5	7.8	85	58	41	28	350	670	15	12	1.0	10
X-3-E	0.035 per cent tallow	0.52	4¾	65	134.9	7.7	5.1	19.4	16.4	47	40	161	6	380	520	99	52	2.9	76
X-3-F	0.07 per cent sulfonated tallow	0.52	3¼	63	139.4	6.8	5.3	17.0	11.3	67	45	145	25	340	555	67	42	1.8	103
X-3-J	0.02 per cent waste sulfite liquor	0.49	3¾	52	130.8	11.8	5.0	21.7	9.4	74	32	156	24	285	450	81	38	2.1	75
X-3-K	0.025 per cent sod. laurel sulfate	0.49	6	66	131.3	9.4	5.0	21.0	16.0	54	41	161	11	325	545	88	44	3.6	...
X-3-L	0.1 per cent hydroxylated organic salt	0.53	2¾	53	147.2	1.7	5.6	11.6	8.9	94	72	22	26	365	760	7	7	1.9	22

CEMENT NO. 2, NOMINAL 7-BAG MIX																			
Y-2-A	None	0.47	2½	41	146.3	2.3	7.0	11.1	10.7	90	89	72	25	380	745	23	23	1.7	68
Y-2-B2	0.04 per cent proprietary resin	0.43	5½	72	134.6	10.4	6.5	18.6	15.3	60	50	160	11	500	535	90	67	2.2	...
Y-2-C	0.04 per cent fish-oil stearate	0.43	3½	57	140.9	5.9	6.8	12.3	12.4	81	78	160	0	635	740	99	85	1.2	...
Y-2-D	0.07 per cent amm. stearate	0.46	4	56	147.2	1.7	7.1	10.7	11.6	95	...	50	25	440	800	20	18	2.7	40
Y-2-E	0.035 per cent tallow	0.46	5	66	142.5	4.4	6.8	12.6	13.1	80	83	137	22	355	670	60	41	1.4	...
Y-2-F	0.05 per cent sulfonated tallow	0.44	5½	59	146.2	2.8	7.0	10.3	11.7	90	92	160	10	550	790	89	74	1.6	...
Y-2-J	0.02 per cent waste sulfite liquor	0.43	3¼	60	140.0	6.1	6.7	11.5	10.6	89	82	160	14	505	755	93	68	1.2	...
Y-2-K	0.025 per cent sod. laurel sulfate	0.45	6	75	140.3	6.5	6.7	16.0	14.8	74	69	161	2	410	675	96	55	2.4	...
Y-2-L	0.1 per cent hydroxylated organic salt	0.44	4	59	148.0	2.2	7.1	10.8	10.9	89	90	160	12	590	750	95	79	1.5	...
CEMENT NO. 3, NOMINAL 7-BAG MIX																			
Y-3-A	None	0.44	6	60	147.2	1.7	7.1	10.3	8.5	95	78	29	28	390	760	10	9	1.7	22
Y-3-B1	0.02 per cent proprietary resin	0.43	3¾	52	140.7	6.1	6.8	15.5	9.3	87	52	160	6	505	640	93	66	1.5	23
Y-3-B2	0.04 per cent proprietary resin	0.43	4½	53	129.7	11.9	6.2	22.8	20.1	47	42	160	7	315	420	90	41	10.6	39
Y-3-C	0.035 per cent fish-oil stearate	0.41	4	55	136.5	7.5	6.6	13.9	8.8	62	39	161	16	440	565	85	58	1.5	...
Y-3-D	0.1 per cent amm. stearate	0.43	3	52	146.8	2.5	7.1	10.7	6.7	90	59	61	28	350	760	22	18	1.1	9
Y-3-E	0.035 per cent tallow	0.43	3¾	63	136.8	7.9	6.6	16.3	11.9	58	42	161	15	390	625	91	51	1.7	23
Y-3-F	0.07 per cent sulfonated tallow	0.42	3½	52	141.3	5.7	6.8	22.6	8.0	79	28	150	25	460[h]	710	64	57	1.1	11
Y-3-J	0.02 per cent waste sulfite liquor	0.41	2¾	46	135.7	9.2	6.6	18.3	8.7	78	37	161	7	385	585	96	51	1.7	20
Y-3-K	0.025 per cent sod. laurel sulfate	0.40	5	61	134.8	9.7	6.5	18.5	12.0	67	43	127	24	290	560	56	30	2.4	...
Y-3-L	0.1 per cent hydroxylated organic salt	0.40	3½	52	148.1	1.9	7.2	9.7	7.2	95	71	120	26	425	795	35	42	1.4	40

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[a] *Proceedings*, Am. Concrete Inst., Vol. 38, November, 1941, p. 121.

[b] *Journal of Research*, Nat. Bureau Standards, December, 1940, p. 711 (RP1349).

[c] $\frac{\text{Bulk Volume} - \text{Solid Volume}}{\text{Bulk Volume}} \times 100.$

[d] $\frac{\text{Volume Water Absorbed}}{\text{Bulk Volume}}.$

[e] $\frac{\text{24-hr. Absorption}}{\text{Total Absorption}} \times 100.$

[f] $\frac{\text{24-hr. Absorption}}{\text{Bulk Volume} - \text{Solid Volume}} \times 100.$

[g] *Proceedings*, Am. Concrete Inst., Vol. 40, June, 1944, p. 573.

[h] One specimen only.

$$f = \frac{qT}{AP}$$

where:

q = number of cubic centimeters of air passing per second through the specimen,

P = pressure difference across the specimen in grams per square centimeter,

A = cross-sectional area in square centimeters over which the flow takes place, and

T = thickness of the specimen in centimeters through which the flow takes place.

The air flow was continued for 10 to 20 min. until the rate had reached apparent equilibrium. Readings were then taken, the air pressure was changed, and when apparent equilibrium was again attained, a new set of readings was taken. Three such observations were taken and averaged.

Other Test:

The resonant frequency determinations were made with the usual equipment for that purpose (7). The specimens were always permitted to remain overnight in the thawing bath before testing since there was a perceptible change in resonant frequency with time immediately following a freezing-and-thawing cycle. By permitting the specimens to remain in the thawing bath overnight the frequency became essentially stable.

The determination of absorption was the last test made. This was conducted in accordance with the A.S.T.M. Standard Methods of Sampling and Testing Brick (C 67–44)[5] except that the specimens were air dried only. On the assumption that all the pores were filled by the absorbed water the porosity was expressed as the ratio of the absorbed water to the bulk volume of the specimen.

DISCUSSION OF RESULTS

Table I summarizes the data obtained in the investigation. The air contents of the concretes varied over a wide range as did the specific weights, the cement factors, and the 35-day flexural strengths. Variations in air-content showed a reasonable correlation with variations in flexural strength as indicated by Fig. 1, and this correlation was best for the rich mixes. The reduction in strength averaged 3.3 and 4.1 per cent, respectively, for each 1-per cent increase in air content for the two cements in the rich mixes, which is in fair agreement with the value of 2 to 3 per cent cited by Gonnerman (1).

The data given in columns *9* and *12*, Table I, which involves use of solid volumes determined by the air-expansion volumenometer, are considered to be lacking in significance for cement No. 2, since the measurements were not made at a constant temperature.

Figure 2 shows the relation for the cement No. 3 concretes between the measured air content of the plastic concrete and the percentage porosity of the hardened concrete as determined by the air-expansion method. The correlation indicated for a given type of concrete is somewhat surprising in view of the number of factors affecting the values plotted, such as errors in methods of measurement, the stability of the air-entraining foam, the large variation in mixing water used, and the fact that one set of measurements was on a plastic concrete and the other on hardened concrete.

A comparison of columns *9* and *10* of Table I (again attention is directed principally to cement No. 3 concretes) shows that a greater porosity was found

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[5] 1944 Book of A.S.T.M. Standards, Part II, p. 178.

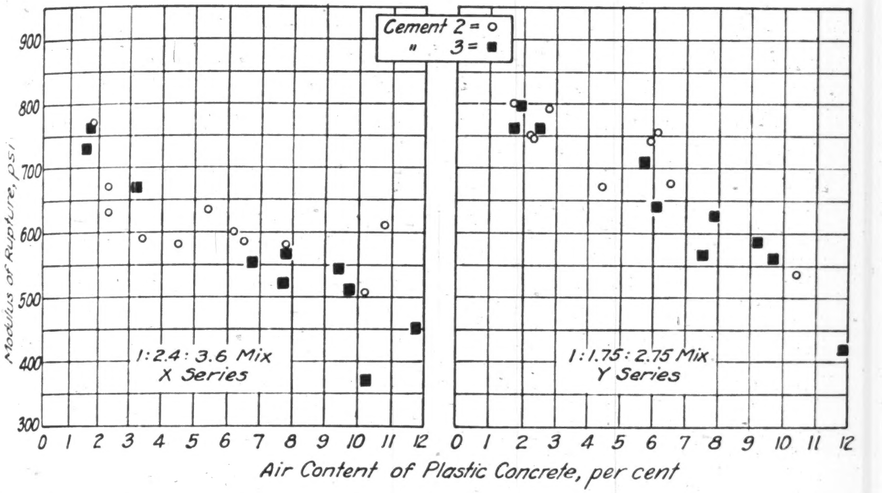

FIG. 1.—Relation Between Air Content of the Plastic Concrete and the 35-day Modulus of Rupture in Bending of Cured Specimens.

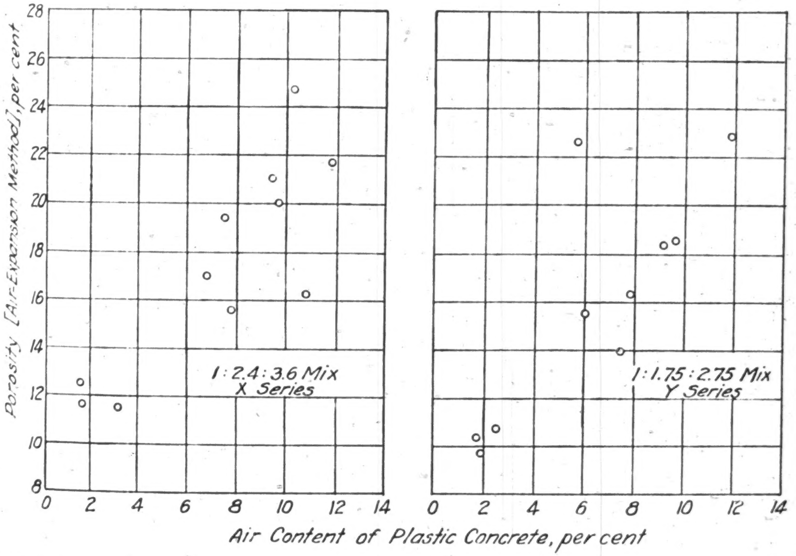

FIG. 2.—Relation Between the Air Content of the Plastic Concrete and the Porosity of the Hardened Concrete Made with Cement No. 3.

by the air-expansion volumenometer method than that calculated from the total absorption. In some cases the difference was very large. Apparently there is little relation between the pore space in the concrete and the absorption determined by the method employed.

The saturation coefficient is usually defined as the ratio of the easily fillable pores to the total fillable pore space. In this case the amount of water absorbed in 24-hr. immersion at room temperature was taken as the measure of the easily fillable pore space. In calculating the saturation coefficients listed in column *11* of Table I the total fillable pore space was taken as the total volume filled by water in the absorption test (5-hr. boil) and the coefficients in column *12* are based on the total pore space as measured by the air-expansion volumenometer method. The differences in porosities as determined by the two methods discussed in the preceding paragraph are again reflected in the wide differences between saturation coefficients.

Columns *14* and *15* give observed data from the freezing-and-thawing tests, and columns *17* and *18* give the calculated durability factors. The dynamic modulus durability factors (*DFE*) and the strength durability factors (*DFS*) are in complete agreement as to which concretes have the poorest durability, and in very good agreement on the concretes having the highest durability. The spread in values between the best and the poorest, however, is much greater when expressed as *DFE* factors than it is when expressed as *DFS* factors. That is to say, some of the specimens that lasted the full 160 cycles with little decrease in dynamic *E* nevertheless had low strengths compared to the unfrozen reference concretes.

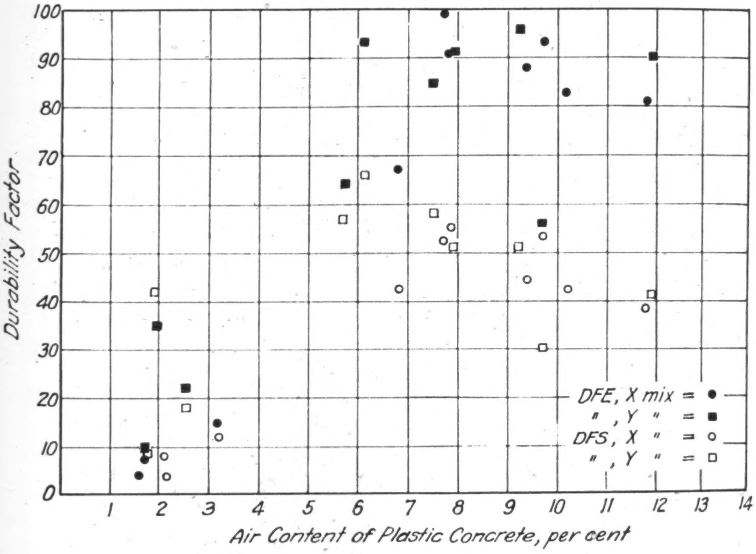

FIG. 3.—Relation Between Air Content of the Plastic Concrete and the Durability Factors of Concretes Made with Cement No. 3.

In most cases the durability factors were higher for the rich concrete than for the corresponding lean concrete. The greatest single difference between the two mixes appeared with the concrete containing admixture L. This admixture, which is not an air-entraining agent but a mild water-reducing agent, increased the density of the concrete. In the lean concrete the durability factor was not significantly affected by the use of this admixture, but in the rich mix, the durability factor was markedly increased.

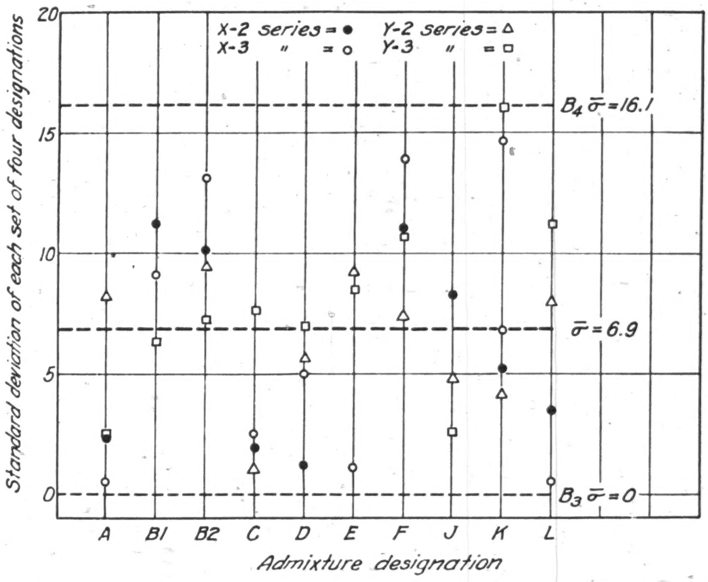

FIG. 4.—Control Chart for Standard Deviations of Durability Factors. (For factors used in computing control chart limits see Table I, Supplement B of A.S.T.M. Manual on Presentation of Data, 1940.)

A comparison between air contents of the plastic concrete and the durability factors for cement No. 3 (Fig. 3) show that the average factor for concretes with less than about 3 per cent air content is appreciably lower than the average factor for concretes containing more than about 6 per cent of entrained air. With air contents above about 6 per cent the strength-durability factors gradually decrease, but the dynamic modulus factors show little or no decrease. The corresponding data for cement No. 2 show the same general trend, though with greater scattering of points.

In studying the data presented in Table I, numerous graphs were made in which the durability factor was the ordinate and one of the following properties was the abscissa: saturation coefficient, total pore space, the ratio of absolute volume of the cement to the volume of the paste, and various other ratios involving the absolute volumes of cement, water, paste and air content. Some relationships were indicated, but none sufficiently quantitative to permit the calculation of one property from another.

The freezing-and-thawing tests were made on four specimens from each batch of concrete. The variations of the durability factors within each set of four specimens should reflect both the precision of the test procedure and the conformity of the test specimens. In making a statistical analysis of the data, the standard deviation was used as a measure of this variation and the "control chart" method as presented in supplement B of the A.S.T.M. Manual on Presentation of Data was used to determine whether the variations in the standard deviation of the various sets was greater than should be attributed to chance. The average standard deviation of all sets $\sigma = 6.9$, determined the central line of the chart and the control limits were calculated as 0 and 16.1. The resulting control chart is shown in Fig. 4. Since all the points fall within the control limits, it is indicated that a state of statistical control existed so far as the conformity of the specimens and the test procedure is concerned.

Referring to Table I and the air-permeability data given in columns *19* and *20*, it is evident that no significant difference was indicated between the air permeabilities of the plain and the air-entrained concretes. The rich concretes had slightly lower average permeabilities than the lean concretes. There was, however, a very large increase in air permeability after the specimens had been tested in freezing and thawing. This increase appears to be unrelated to durability factor.

A value for the mean effective capillary radius can be calculated from the permeability and porosity data if it is assumed that the pore system is made up of N identical parallel, uniformly distributed cylindrical capillary passages with a radius r and length T and that this system has the same air permeability as the actual specimen.

The equations as given by Stull and Johnson (4) are:

$$r = 0.012\,(f/V)^{1/2}$$
$$N = 21.7\,(V^2/f)$$

where:

r = mean effective capillary radius in centimeters,
N = number of capillaries of radius r, per square centimeter of surface,
f = air permeability, and
$V = 100\,(\pi r^2 N)$ = percentage porosity.

The values calculated for r for the specimens tested in this investigation ranged from approximately 0.1 μ to 0.3 μ. As with the values for air permeability, no trends were indicated with respect to air entrainment, but there was slight evidence that the richer concretes had pores of smaller average radius than those in the lean concretes.

TABLE II.—RELATION BETWEEN THE RATIO OF AIR TO WATER PERMEABILITY AND THE MEAN CAPILLARY RADIUS (OF CLAY BRICKS).

(From R. T. Stull and P. V. Johnson (4).)

Series	Number of Bricks	Ratio of Air Permeability to Water Permeability	Ratio of Air Permeability to Water Permeability × 55.5	Mean Capillary Radius, μ
F	6	572	10.3	1.37
G	11	481	8.7	1.64
H	10	410	7.4	2.97
K	10	184	3.3	3.28
J	10	169	3.0	4.13

According to Poiseuille's equation, the permeability of a material to two fluids should be inversely proportional to the respective viscosities of the fluids. It has been shown, however, by Mills (8) and by Stull and Johnson (4) that with brick the permeabilities to water and to air are not inversely proportional to the viscosities of water and of air. Stull and Johnson give data, reproduced in part in Table II, indicating that as the calculated average pore diameter increases the respective permeabilities approach the theoretical ratio.

An extrapolation of the data in Table II indicates that the ratio of air permeability to water permeability should reach the theoretical value of unity (air permeability = water permeability × 55.5) at a capillary radius of about 5

μ. Since the average capillary radius for the concrete as determined in this investigation was much less than any of the values given in Table II for brick (the r values were calculated by the same formula in both cases) it is indicated that for concrete the ratio of air permeability to water permeability also will not be inversely proportional to the respective viscosities.

Ruettgers, Vidal, and Wing (9) have demonstrated in tests of water permeability that end effects (skin effects) may offer hydraulic resistance equivalent to 3 to 6 in. of normal concrete. Accordingly it is quite possible that, with the specimens tested ($2\frac{1}{16}$ in. thick), the end effects masked effects of such factors as air entrainment and richness of mix that ordinarily might affect permeability.

SUMMARY

Results are reported of tests on concretes of two proportions, each made with cements of two series. The cements were made from two different clinkers by grinding successive portions of the clinkers without added admixture (plain cements) or by intergrinding in turn each of six admixtures principally of the air-entraining type. Also, two admixtures were added to the plain cements at the time of mixing in the concrete mixer.

The air contents of the concretes varied over a wide range and, since no adjustments were made in the mix to compensate for the bulking of the entrained air, there was a wide variation in cement factors.

The pore space in dried concrete was determined by means of an air-expansion volumenometer. Water absorptions of the same specimens were determined by 24-hr. immersion at room temperature (24-hr. absorption) followed by immersion in boiling water for 5 hr. and cooling in the water (total absorption). Restricting consideration to concretes made with cement No. 3, because much better laboratory control was exercised in volumenometer tests with this cement, the percentage porosity determined by the air-expansion method was higher in nearly all cases than porosity calculated from total absorptions. Similarly, the saturation coefficients calculated from the ratio of the 24-hr. absorption to the total absorption were higher, in most cases, than the coefficients calculated from the ratio of 24-hr. absorption to pore space determined by the air-expansion method. It was indicated accordingly, that the method employed of determining absorption lacked considerable of filling all the pore space with water.

Specimens were subjected to repeated cycles of freezing and thawing (fast cycle) and the resulting durability factors were calculated.

The durability factors of the concretes were, with one exception, materially lower for concretes containing less than about 3 per cent by volume total air, than for concretes containing about 6 per cent or more.

The air permeabilities for all except two of the concretes ranged from approximately 1×10^{-5} to approximately 6×10^{-5} cu. cm. per sec. per cu. cm. of concrete per gram per sq. cm. pressure difference. The two concretes constituting the exception had much higher air permeabilities than the other concretes and also had the highest total absorptions of all the concretes.

The air permeabilities of specimens which had been subjected to freezing tests were considerably higher than for those not subjected to freezing. This was true even for those specimens that had high durability factors.

No trends in air permeabilities were apparent with respect to air entrainment. The concretes from the rich mix had slightly lower average air permeabilities than the lean concretes. The specimens tested were small, however, and end effects may have masked some effects.

REFERENCES

(1) H. F. Gonnerman, "Tests of Concretes Containing Air-Entraining Portland Cements or Air-Entraining Materials Added to Batch at Mixer," *Proceedings*, Am. Concrete Inst., Vol. 40, June, 1944, p. 477.

(2) Charles E. Wuerpel, "Laboratory Studies of Concrete Containing Air-Entraining Admixtures," *Proceedings*, Am. Concrete Inst., Vol. 42, February, 1946, p. 305.

(3) George L. Pigman, "A Vacuum Method of Measuring the Air Content of Fresh Concrete," *Proceedings*, Am. Concrete Inst., Vol. 38, November, 1941, p. 121.

(4) Ray T. Stull and Paul V. Johnson, "Some Properties of the Pore System in Bricks and Their Relation to Frost Action," *Journal of Research*, Nat. Bureau Standards, Vol. 25, December, 1940, p. 711 (RP1349).

(5) Stanton Walker, "Freezing-and-Thawing Tests of Concrete Made With Different Aggregates," *Proceedings*, Am. Concrete Inst., Vol. 40, June, 1944, p. 573.

(6) Joseph C. Richmond, John B. Petersen, and Winslow H. Herschel, "An Improved Volumenometer," *Journal*, Am. Ceramic Soc., Vol. 26, No. 4, April, 1943, p. 127.

(7) Leonard Obert and Wilbur I. Duvall, "Discussion of Dynamic Methods of Testing Concrete with Suggestions for Standardization," *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 1053 (1941).

(8) E. O. Mills, "The Permeability to Air and to Water of Some Building Bricks," *Transactions*, British Ceramic Soc., Vol. 33, p. 210 (1933–34).

(9) Arthur Ruettgers, E. N. Vidal, and S. P. Wing, "An Investigation of Permeability of Mass Concrete with Particular Reference to Boulder Dam," *Proceedings*, Am. Concrete Inst., Vol. 31, p. 382 (March, April, 1935).

PERMEABILITY OF BRICK-MORTAR ASSEMBLAGES*

By John W. McBurney,[1] M. Arnold Copeland,[1] and Russel C. Brink[1]

Synopsis

Water-permeability tests were made at 35 days and again after 1 and 2½-yr. exposure on brick-mortar assemblages constructed from ten types of brick and three types of mortars (A, B, and C). The 2½-yr. permeability test was made with water solutions of dyes of contrasting colors. This procedure demonstrated extent of bond between brick and mortar and permitted an estimation of the size and nature of through channels. The initial rate of absorption of the bricks had greatest effect on permeability.

It is well recognized (**1**, **2**, **3**)[2] that the initial rate of absorption of clay and shale building bricks is one of the important factors affecting the water permeability of brick-mortar combinations in masonry. The present paper may be considered as an extension of the work of Fishburn and his co-workers (**1**, **2**) and of Palmer and Parsons (**3**) in that additional properties of bricks are determined and reported. The effect of exposure to weather on the permeability of the test panels was observed up to 2½ yr., and some information on the nature and extent of the contact between brick and mortar was obtained by the use of a modification of Anderegg's dye test (**4**).

Materials and Methods of Testing of Materials

Brick:

Nineteen kinds of brick had been selected for use in an investigation of the factors affecting the strength of bond between brick and mortar. This selection was from the standpoint of reasonably representing the range of materials, methods of forming, and properties of brick produced in the United States. Ten of these 19 types were used in the present investigation. Determinations of initial rate of absorption, water absorption by 24-hr. cold submersion, and by 5-hr. boiling, saturation coefficient (*C*/*B* ratio), and compressive and transverse strength were made on 20 specimens of each type, unless otherwise noted. The methods of test were those set forth in the Standard Methods of Sampling and Testing Brick (C 67 – 44)[3].

Porosity and air permeability were determined and the derived "average radii of capillaries" and "average number of capillaries per sq. cm." were calculated by the methods of Stull and Johnson (**5**) on one specimen of each sample. Each of these specimens was selected so that its water absorption by 5-hr. boiling was the median for the sample.

The data on the properties of the brick

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] Technologist and Junior Engineers, respectively, National Bureau of Standards, Washington, D. C.

[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 1348.

[3] 1944 Book of A.S.T.M. Standards, Part II, p. 178.

used in this investigation together with descriptions are given in Table I.

Mortar Materials:

Portland cement was purchased under Federal Specification SS-C-191a. Ten sacks were mixed at a time and stored in air-tight steel drums during use. A second shipment of cement was required before completion of construction of the test specimens, but no significant or consistent differences in behavior of mortars or assemblages were noted.

TABLE I.—IDENTIFICATION AND PROPERTIES OF BRICKS.

Brick	No. 1	No. 3	No. 7	No. 8	No. 9	No. 10	No. 11	No. 14	No. 15	No. 19
Corresponding number in BMS60[a]	225	224	64-2	66-1	70-2A	136	13-6A	26-4	94-1A	228-2
Table in BMS60[a]	10	10	7	7	9	4	12	1	4	25
Method of forming[b]	SC	SC	SM	SC	SM	SM	SM	SM	SM	DP
Material[c]	fc	s	c	c	c	c	c	c	c	c
Description of surface[d]	R	R	ss, I	R	ss, Sm	ss, I	ss, I	ss, I, Sm	ss, I	Sm
Penetrability average[e], g. per min	0.5	8	26[f]	33	33	41	42	58	66	127
Maximum penetrability, g. per min.	2.0	23	36	63	51	59	64	94	81	175
Minimum penetrability, g. per min.	0.0	1	18	17	25	29	21	41	47	89
Water absorption by 24 hr. cold immersion, per cent[e]	0.8	2.4	11.3	9.4	12.6	13.8	20.2	15.5	17.4	15.9
Water absorption by 5-hr. boiling, per cent[e]	2.2	4.4	15.1	13.4	17.3	18.7	23.5	18.5	21.1	18.6
Saturation coefficient C/B[e]	0.34	0.55	0.74	0.70	0.73	0.74	0.86	0.84	0.83	0.86
Porosity (V), per cent	6.4	12.4	....	29.6	33.1	....	43.2	38.4	40.9	29
Air permeability ($f \times 10^5$)[h]	48	1750	....	2020	805	....	102	72	180	2200
Capillary radius, micron[h]	1.05	4.50	....	3.15	1.90	....	0.60	0.50	0.80	3.30
No. of capillaries per sq. cm. ($N \times 10^{-4}$)[h]	185	19	....	94	300	....	3930	4420	2000	89
Compressive strength, psi.[e, i]	15 400	13 400	2670[j]	5360[j]	5370	3240[j]	3640	4500	3730	3180
Modulus of rupture, psi.[e]	2765	1 685	550[i]	820[i]	860	540	565	650	465	250

[a] J. W. McBurney and J. C. Richmond, "Strength, Absorption and Resistance to Laboratory Freezing and Thawing of Building Bricks Produced in the United States," BMS60 (Nov. 1940).
[b] Methods of forming: SC—side cut; SM—soft mud; DP—dry press.
[c] Materials: fc—fire clay; s—shale; c—clay.
[d] Description of surfaces: s—sand struck; R—rough; I—intermediate; Sm—smooth.
[e] Average of 20 tests unless otherwise noted.
[f] Average of 30 tests.
[g] A resample of this shipment gave 37 g. per minute for the average.
[h] Test on one specimen selected for average absorption.
[i] Average from BMS60, unless otherwise noted.
[j] Average from Fishburn's data.

Lime putty was made from quicklime corresponding to the lime A previously described (6), and was aged at least two weeks before use. Lime A is a high-calcium quicklime and produces a putty with excellent plasticizing properties. The putty was analyzed from time to time for content of hydrated lime and the quantities used were adjusted to supply the required weights of hydrated lime in the mortars.

Potomac River mason's sand was used after oven drying. A typical sieve analysis is as follows:

Sieve	Percentage Passing
No. 4	100
No. 8	99.8
No. 16	98.4
No. 30	84.6
No. 50	27.0
No. 100	6.1

This gives a fineness modulus of 1.84.

Mortars:

Three mortar mixtures were used corresponding to the A, B, and C mortars of the American Standard Building Code Requirements for Masonry,[4] and are hereinafter referred to by these letters. The weight proportions were 94:9.4:282 (cement, equivalent hydrated lime, dry sand) for mortar A; 94:40:470 for mortar B; and 94:80:705 for mortar C. Assuming 94, 40, and 94 as the weight per cubic foot of portland cement, hydrated lime, and dry sand, respectively, these three mixtures correspond to 1:0.25:3.

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[4] Nat. Bureau Standards Miscellaneous Publication M174, U. S. Government Printing Office, March 15, 1944

1:1:5, and 1:2:7.5, proportions by volume.[5]

Ten-kilogram batches of dry ingredients were mixed at a time and water was added to give an initial flow between 100 and 110 per cent. Water retentivity was determined immediately after mixing, and briquettes and 2-in. cubes were cast in triplicate for tension and compression tests. Water retentivity was again determined after completing construction of a set of three panels. Initial flow and water retentivity were determined by methods conforming to those used for testing masonry cements.[6]

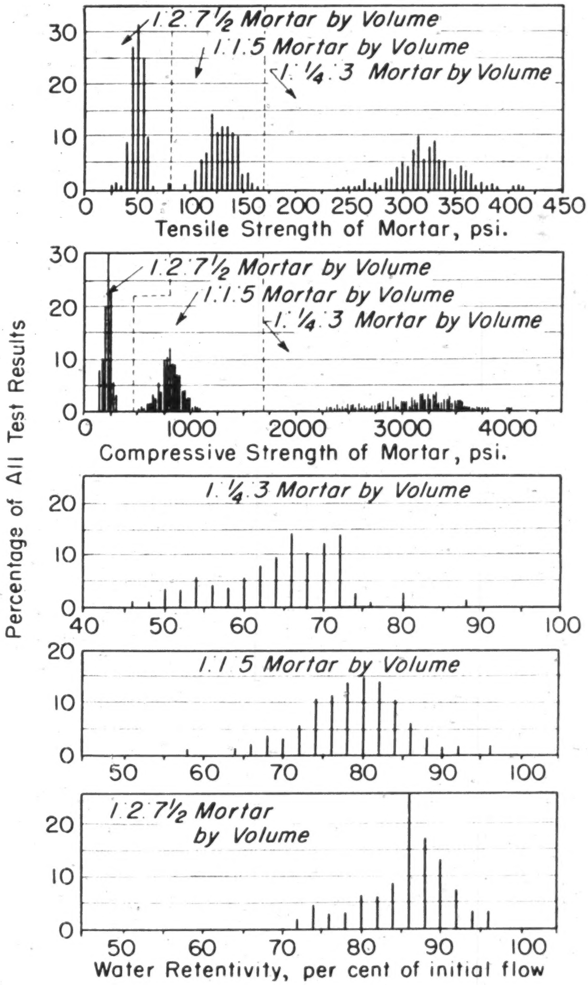

FIG. 1.—Frequency Distributions of Results of Tension and Compression Tests and Water Retentivities of $1:\frac{1}{4}:3$ (Mortar A), 1:1:5 (Mortar B), and $1:2:7\frac{1}{2}$ (Mortar C) Cement-Lime-Sand Mortars by Volume.

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[5] Since a cubic foot of damp, loose sand contains approximately 80 lb. of dry sand, the volume proportions of mortars A, B, and C correspond to 1:0.25:3.5, 1:1:5.9, and 1:2:8.3, respectively, on the basis of commercial practice using damp sand.

[6] Standard Specifications for Masonry Cements (C 91 – 40), 1942 Book of A.S.T.M. Standards, Part II, p. 7.

TABLE II.—RESULTS OF TESTS OF MORTAR.

Nature of Test	Mortar A			Mortar B			Mortar C		
	Average	Maximum	Minimum	Average	Maximum	Minimum	Average	Maximum	Minimum
Water retentivity, per cent	64	88	46	78	96	58	85	96	72
Compressive strength, psi	3090	4025	2225	820	1100	525	230	300	150
Tensile strength, psi	330	415	240	130	165	85	50	80	25

Except for the method of curing (one day in molds and 27 days in water), compressive strengths were also determined as for masonry cement.[6] Tensile strengths were determined at 28 days by the method used for portland cement.[7]

The average, maximum, and minimum values of these tests are given in Table II.

The distributions of individual values are presented in Fig. 1. These data are discussed in a later section.

Construction of Panels

Permeability test panels were constructed with five whole-bricks and two half-bricks, as shown in Fig. 2. The ends of the bricks were heavily "buttered" with mortar. The mortar bed joint was spread over the two-brick length and the bricks of the next course were placed with a shoving motion. Bricks with "frogs" were laid frog up. One end of the panel abutted on a vertical stop to give alignment of the ends of the bricks and to prevent displacement of bricks previously placed. The mortar joints were "struck" on one face of the panel and "tooled" on the other. Three panels were constructed from each batch of mortar (10 kg.). Unless otherwise indicated, all bricks were oven-dried before laying. Where wetted bricks were used, the procedure was to submerge the bricks in water at room temperature for 30 min. and then stand them on end on a concrete floor for 30 min. in the laboratory. Construction of the panels started immediately following this drying period.

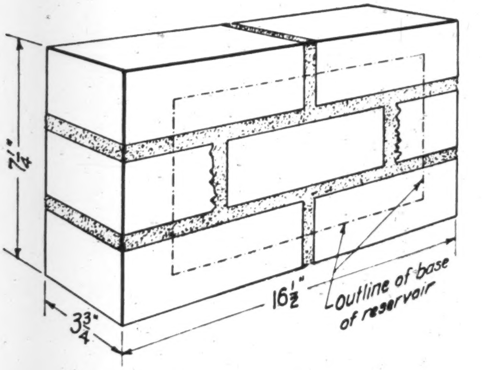

Fig. 2.—Diagram of Specimen Used for Permeability Tests with Water and Dye Solutions.

The panels were built in a room where the temperature was maintained at 70 ± 2 F. with relative humidity of 80 ± 2 per cent and were left undisturbed for 24 hr. They were then stored for 27 days in a spray room (70 ± 2 F., relative humidity 100 per cent) and were afterwards dried for 7 days in a room kept at approximately 100 F. before being tested for permeability.

Testing of Panels

The dried panel was laid on its side with the "tooled" face up. A rectangular impounding reservoir 6 by 12 by 1½ in. deep was built on the face of the panel by bending a strip of sheet metal to the proper shape, soldering the ends of the strip together, and attaching to the panel with a plastic calking compound (see Fig. 2). This is the method used by Palmer and Parsons (3). The panel was then supported over a drip pan and a 1-in. head of water was maintained in the impounding reservoir during test. The observations and measurements included: (1) noting location and time of appearance of first damp spot on the lower face of the panel; (2) location and time for the second damp spot; (3) noting time for appearance of first drop of water; and (4) measuring quantity of water (if any) in drip pan after 4 min., 1 hr., and 2 hr. If a measurable quantity of water was not obtained within 2 hr., the test was continued up to 48 hr. No corrections were made for evaporation of water from the drip pan. The rate of evaporation would obviously depend upon conditions, such as temperature, relative humidity, and wind velocity. One test, made without record of conditions, during hot weather, indicated evaporation at the rate of 7 ml. per hour.

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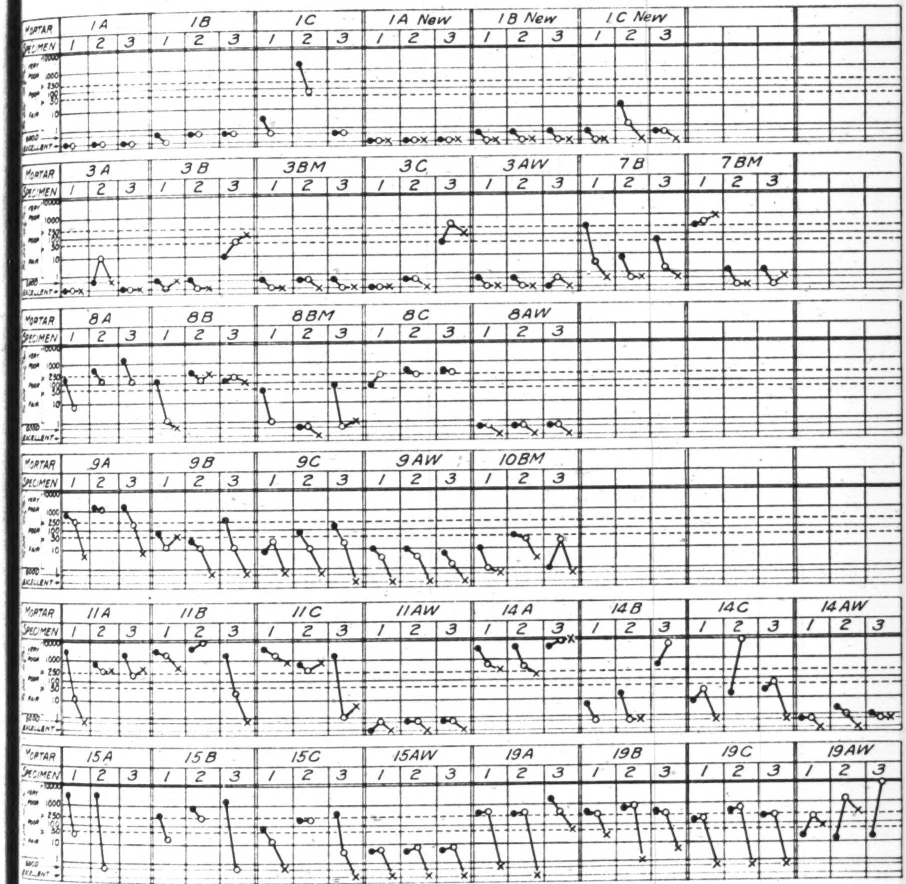
[7] Standard Methods of Sampling and Testing Portland Cement (C 77 – 40), 1942 Book of A.S.T.M. Standards, Part II, p. 33.



NOTE: ● = Test at 35 days, ○ = test at 1 year and × = test at 2½ years.
Numbers refer to brick, letters A, B and C identify mortars. M= machine mixed mortar, W= wetted brick.

FIG. 3.—Results of Permeabilty Tests in Terms of Ratings of "Excellent" (No Dampness or Leakage), "Good" (Dampness Only), and Flow as Milliliters per Hour.

After completion of the test for permeability (age of specimen 35 days, and hereinafter called "initial permeability"), the panels were exposed on the roof of one of the Bureau buildings for approximately 1 yr. then redried, and retested up to 24 hr. by the same procedure. During exposure, the panels stood upright on the slag surface of the roof. The drainage was such that no pools of water were formed.

A third permeability test was made after approximately $2\frac{1}{2}$ yr. of exposure. A water solution of a blue dye (diazine blue) was used for 2 hr., followed by a 10-min. permeation with a water solution of a yellow dye (metanil yellow).[8] After treatment with these dye solutions, the panels were dried and then taken apart. All mortar and brick interfaces were examined for presence, depth of penetration, and color of stain, and the results recorded diagrammatically. The interpretation of the results of permeation with dyes of contrasting colors is discussed in a later section.

Presentation of Data on Tests of Panels

The results of the 35-day, 1-yr., and $2\frac{1}{2}$-yr. permeability tests in terms of rate of flow (milliliters per hour) are presented in Fig. 3. Tables have been prepared presenting these data as well as results for "time for first dampness" and "time for first drop," but the tabular presentation is omitted on account of limitations of space.

Figure 3 uses the nomenclature of Fishburn's classification (**1, 2**), but is based upon the following ratings.

Excellent (E).—No dampness on lower face of specimen for duration of test (48 hr. for first, 24 hr. for second, and 2 hr. 10 min. for third test). It will be noted that the third test is indeterminate in comparison with the 48 and 24-hr. tests, unless dampness or leakage occurred within the interval of the test (2 hr. 10 min.). Some consideration was given to "adjusting" the observation on the third test to allow for behavior observed after 2 hr. in the longer tests, but this was not done.

Good (G).—Dampness without leakage for duration of test (48, 24, and 2 hr. 10 min.-tests). The rating for the short-time test is likewise indeterminate, if leakage occurred after 2 hr. in the first and second tests.

Fair (F).—Leakage not exceeding 50 ml. in 1 hr.

Poor (P).—Leakage not less than 50 or more than 250 ml. in 1 hr.

Very poor (VP).—Leakage at a rate exceeding 250 ml. in 1 hr.

Characteristic dye-test diagrams are shown in Figs. 4, 5, and 6.

Discussion of Data

Workmanship:

The intention was to eliminate workmanship as a factor by using the best possible degree of joint filling and by following a uniform technique. Under these conditions, differences in results would be ascribed to differences in properties of the materials. Two operators built panels and each operator's construction included panels made with bricks of low as well as high rates of absorption. For each particular brick, all panels were made by the same operator, except that duplicate sets of panels of brick 1 were made by two operators. The excellent agreement in the results of tests for brick 1, laid with mortars A and B by these two operators, will be noted. In agreement, also, is the erratic nature of the results from both operators using brick 1 laid with mortar C.

Brick:

The following discussion is intended to call attention to certain peculiarities of the brick samples used in these tests and to amplify some of the data presented in Table I.

Roughness of surface.—If it be assumed that the nature of the brick surface in contact with the mortar affects the

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[8] The use of two dyes of contrasting colors was suggested and developed by M. A. Copeland.

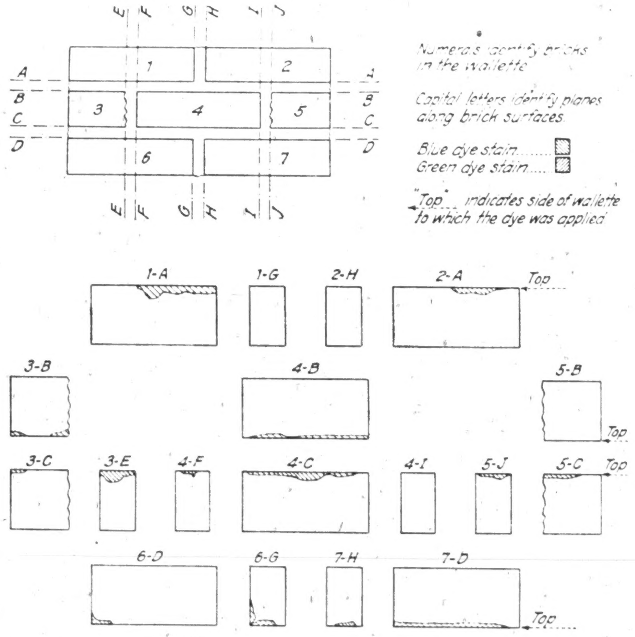
E F G H I J
1
2
3
4
5
6
7
A
B
C
D
Numerals identify bricks in the wallette.
Capital letters identify planes along brick surfaces.
Blue dye stain.......
Green dye stain.....
"Top" indicates side of wallette to which the dye was applied.
1-A
1-G
2-H
2-A
Top
3-B
4-B
5-B
Top
3-C
3-E
4-F
4-C
4-I
5-J
5-C
Top
6-D
6-G
7-H
7-D
Top

FIG. 4.—Diagram of Results of Dye-Permeability Test on Panel Built with Brick No. 3 and Mortar A (Specimen 3A1).

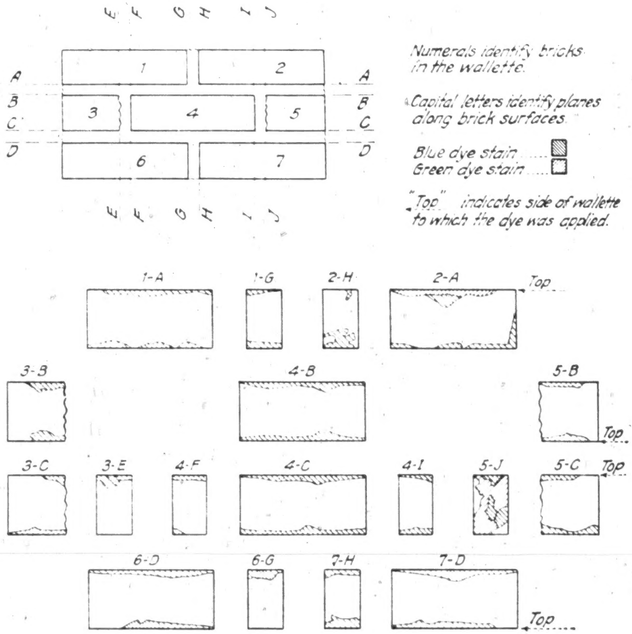
E F G H I J
1
2
3
4
5
6
7
A
B
C
D
Numerals identify bricks in the wallette.
Capital letters identify planes along brick surfaces.
Blue dye stain.......
Green dye stain.....
"Top" indicates side of wallette to which the dye was applied.
1-A
1-G
2-H
2-A
Top
3-B
4-B
5-B
Top
3-C
3-E
4-F
4-C
4-I
5-J
5-C
Top
6-D
6-G
7-H
7-D
Top

FIG. 5.—Diagram of Results of Dye-Permeability Test on Panel Built with Brick No. 3 and Mortar C (Specimen 3C3).

permeability of the resulting brick-mortar interface, some amplification of the data given for "Description of Surface" in Table I appears justified. There are types as well as degrees of roughness. Among the types are: (1) the rough "hackly" surface produced by wire cutting (bricks 3, 1, and 8 in decreasing order of roughness); (2) a second type of roughness is provided by sand-struck, soft-mud brick. (The apparent mechanical roughness depends upon the size of the sand grains and permeability may be governed by the degree of adherence of the sand to the brick surface. The sand-struck surface is usually the frogged flat. The other flat shows little sanding, but is usually mechanically roughened by "striking off" the clay in the mold.) Listed in order of decreasing size of sand grain are 14, 15, 7, 9, 10, and 11. (3) The dry-press sample (brick 19) has very smooth and regular flats. This brick's rather large "average radius of capillaries" might be considered as giving it a micro roughness. Large capillary radii also characterize bricks 3 and 8 according to the measurements.

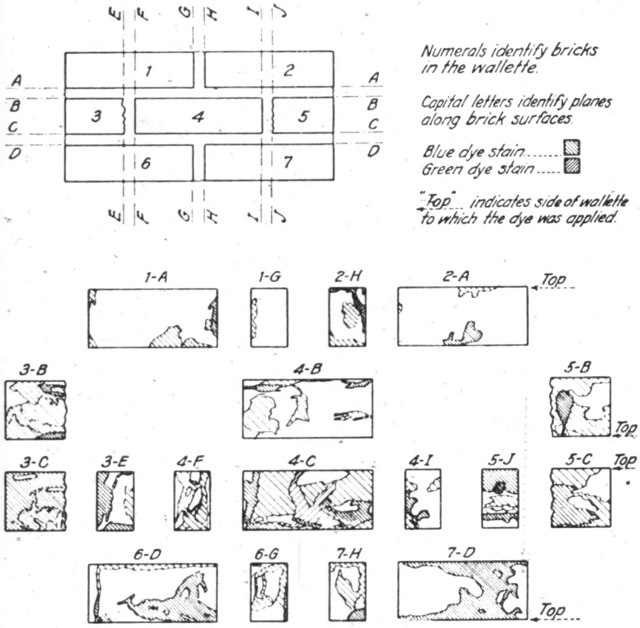
E F G H I J
A B C D
1 2 3 4 5 6 7
Numerals identify bricks in the wallette.
Capital letters identify planes along brick surfaces.
Blue dye stain ▨
Green dye stain ▨
"Top" indicates side of wallette to which the dye was applied.
1-A 1-G 2-H 2-A
3-B 4-B 5-B
3-C 3-E 4-F 4-C 4-I 5-J 5-C
6-D 6-G 7-H 7-D
Top

Fig. 6.—Diagram of Results of Dye-Permeability Tests on Panel Built with Brick No. 11 and Mortar C (Specimen 11C1).

No end-cut brick or water-struck, soft-mud brick were included in this program. It can be stated, however, on the basis of the brick that were used, that differences in roughness did not appear to be related to degree of permeability. The effect, if any, was masked by the effect of differences in other properties.

Relations Among Properties.—It will be noted in Table I that the bricks are listed in order of increasing penetrability (initial rate of absorption). It will also be noted that, in general, the two immersion absorptions, the saturation coefficients, and the porosities tend to increase with increase in penetrability. Compressive and transverse strength, however, tend to decrease with increase in these other properties. It follows, therefore, that the relations of but one of these properties to permeability need be considered in detail. The property selected for consideration is penetrability. (While it is true that compressive strength, for example, is inversely related to degree of permeability, it is incorrect to infer that the relation is causal.)

Permeability to air is poorly related to the properties listed above. The "average radii of capillaries" and "number of capillaries per square centimeter" (both values derived from porosity and air-permeability values) are inversely related to each other, but are poorly related to all of the other properties.

Brick 3 is unusual in several respects. Its initial rate of absorption, although low (8 g. per minute) is higher than would be expected from its absorption by 5-hr. boiling (4.4 per cent). Its high air permeability, taken with its rather low porosity, gives the very high value of 4.50 microns for "average capillary radius." Laminar cracking is observed on these bricks which provides a possible explanation of these values.

Durability.—BMS60 (7) gives the results of 50 cycles of freezing and thawing on samples of the bricks used in this investigation. Disintegration was observed on specimens in samples with saturation coefficients exceeding 0.80 (bricks 11, 14, 15, and 19). The effect of durability of the bricks on permeability of the panels is discussed under Effect of Exposure on Permeability.

Mortar:

The frequency distributions of the strengths and water retentivities of the mortars used in this investigation (Fig. 1) show greater ranges than are usually recorded. The following considerations may be pertinent.

Two different 10-in. flow tables were used. The readings on these two tables were usually not in agreement and the differences were not constant. One of these tables was assumed to be constant and "correct." The validity of this assumption was never convincingly proved.

The ingredients of the mortars were carefully proportioned, but the mixing may be described as commercial since the mortars were mixed by hand and by machine and by different operators and the procedures prescribed in the A.S.-T. M. methods of test for masonry and portland cement[6, 7] were not followed. (Unpublished data indicate that variations in the technique of mixing samples of mortars change the apparent flows very considerably.) Water retentivities were measured both before and after building a triplicate set of panels, and these differences in the ages of the mortar specimen at time of test may explain in part the scatter shown in Fig. 1.

All three mortars were considered by the several operators to be easily workable and, although new shipments of sand, cement, and lime were secured before completion of the work, no observable differences were reported. The effect of mortars A, B, and C on initial permeability of the panels is discussed later.

Effect of Properties of Bricks on Initial Permeability of Panels:

As stated previously, initial rate of absorption, the two water absorptions, and porosity are all well and directly related to each other, and the two strength measures are equally well but inversely related to the absorption. Since (*a*) there is evidence that initial rate of absorption (penetrability) is the most important of these properties, and (*b*) these other properties would be similarly related, attention will be confined for the most part to the effect of variation in penetrability.

Initial Rate of Absorption.—Reference has been made to an investigation of the effect of properties of bricks on strength of bond between brick and mortar. One of the conclusions from that investigation was that bricks with penetrabilities less than 10 g. per minute gave very high bond strengths both in tension and in shear when laid with mortar A, whereas bricks with penetrabilities exceeding 20 g. per minute gave low bond strengths. This conclusion, based on averages of specimens, was confirmed by the results of tests on a series of specimens built from bricks having selected penetrabilities within ± 1 g. and covering the range from 10 to 20 g. per minute. The relations between penetrability of brick and permeability of panels are as follows: With one exception (specimen 3A2 which may have contained a cracked brick), all panels built with bricks having penetrabilities of less than 10 g. per minute (Nos. 1 and 3) and with mortar A gave zero water permeability (no dampness or leakage in 48 hr.) when tested at 35 days. (The apparently critical range of 10 to 20 g. per minute is not represented in these tests.) The next highest rate (33 g. per minute for brick 8) was associated with permeability results of from 156 to 1070 ml. per hour on panels laid with mortar A. With this same mortar, permeability of the panel increased with increasing penetrability of the brick until, with brick 14 (58 g. per minute), all permeabilities at 35 days exceeded 4000 ml. per hour. With brick 15 (66 g. per minute), one of the panels failed before it could be tested (zero bond between brick and mortar). Brick 19 (127 g. per minute) gave somewhat better results (460 to 1300 ml. per hour). Brick 19 is discussed in the following section.

The conclusion that low initial rates of absorption of bricks at time of laying with mortar A result in low permeabilities to water of the panels is confirmed by the pronounced improvement resulting from laying bricks 8, 9, 11, 14, and 15 wetted instead of dry.

Panels laid with mortar B show the same general trend with regard to effect of initial rate of absorption of the bricks on permeability of the panels. The low-rate-absorption bricks are associated with "good" (dampness only) permeability tests. Leakage becomes evident when bricks having an initial absorption exceeding 25 g. per minute (No. 7) are used, but the rate of leakage is, in general, less than that observed on the corresponding panels laid with mortar A. For the bricks having high initial rates of absorption, the leakage results are erratic, sometimes exceeding and sometimes being less than the corresponding panels with mortar A.

Mortar C is associated with still more erratic results. Leakage ratings from "fair" to "very poor" occur over the entire range of brick penetrabilities. All that can be said is that better leakage ratings occur more frequently when bricks have a low initial rate of absorption and conversely that "very poor" ratings are most frequently associated with bricks having a high initial rate of absorption.

Other Properties of Bricks.—Under the conditions of test (1-in. head of water), it would be expected that water would "strike through" a $3\frac{3}{4}$-in. thickness of all except the most dense and impermeable bricks during a 48-hr. period of test, and therefore that "good" (dampness only) would be the best rating obtained for any panel built with bricks other than 1 and 3. The only exception was provided by a single panel laid with brick 11, wetted. Brick 19 was the only type through which water flow was definitely observed. There was some evidence for considering that water flowed through brick 15, though at a very low rate. An individual specimen of brick 3 which exhibited laminar cracking was recorded as responsible for the high permeability of panel 3C3. The laminar structure of bricks 3 and 8 is a probable explanation of their high air permeabilities with consequent large "average radii of capillaries." The high readings on brick 19 can, however, be rather certainly ascribed to pore structure.

That brick 11 had such very poor permeability ratings with all mortars may be due to its "sand" being more of a dust than a sand. The effect of dusty bricks on bond is well-known. The improvement resulting from wetting these bricks will be noted (see next section).

Effect of Wetting Bricks Before Laying.—The effectiveness of wetting absorbent bricks before laying in reducing the initial permeability of panels built with mortar A is very evident. It will be observed that, without exception, wetting bricks 8 to 19 and laying with mortar A gave better results than were obtained with the use of mortars A, B, and C on the same bricks laid dry. Brick 11 provides an outstanding example of such improvement. For bricks 8, 9, 10, 11, 14, and 15 (average initial rate of absorption from 33 to 66 g. per minute), the permeability results on the panels built with wetted bricks were either "good" (dampness only) or very low rates in the "fair" range (leakage not exceeding 50 ml. per hour).

The failure of bricks 8 to 15 to obtain "excellent" ratings (no dampness or leakage), except in the case of 11AW1, resulted from the fact that water struck through the bricks themselves, with consequent appearance of dampness before finishing the test. The values 15 to 19 ml. per hour for the initial tests on 19AW are very probably the water permeabilities of the brick units.

The initial permeability tests on panels built with brick 3, wetted, were better than the corresponding results using mortars B and C on dry bricks, but not so good as the results determined on the dry-brick-mortar A combination. That dampness occurred on two out of three 3AW panels may possibly be explained by increase in the water content of the mortar from free water on the bricks, with consequent increase in the porosity and permeability of the mortar.

Effect of Properties of Mortar on Initial Permeability of Panels:

The effect of the several mortars has been discussed in some detail in connection with consideration of the effect of brick properties. Some further discussion together with a summary is presented here. An increase of 21 per cent in average water retentivity (mortar C compared with mortar A) materially lowered the rate of flow of panels 8C, 9C, 14C, and 15C compared with 8A, 9A, 14A, and 15A, respectively. With the exception of panels 9C1 and 14C1, 2, and 3 (initial ratings of "fair"), all of the C panels, however, had ratings of "poor" and "very poor" (50 to 250 and over 250 ml. per hour). With one exception (8A1), all of the panels built from bricks 8, 9, 14, and 15 laid dry with mortar A, rated "very poor." Brick 11 is of interest in that no significant change in the very high rate of flow through panels made from this brick, laid dry, was produced by differences in mortars. The type of mortar had little effect also on panels built from brick 19.

Use of mortar C with the two low-penetrability bricks (1 and 3) resulted in definitely poorer performance in comparison with mortar A. The observed permeability of mortar C would almost ensure an observation of dampness for the conditions of test and dimensions of specimens used in this program. Mortar B may be dismissed as being intermediate in results between mortars A and C.

For the particular bricks and mortars used in this investigation, the conclusion appears justified that the properties of the bricks (in particular, initial rate of absorption) have a greater effect on performance in permeability tests than properties of the mortars in the range used. The lime used in these mortars was one of the best plasticizing agents known at the time these tests were scheduled. Use of other limes, such as the recently developed pressure hydrates, might improve performance. On the other hand, use of lime C (6) might have the effect of emphasizing the importance of mortar by giving poorer results. Masonry cements might be selected to give a range in water retentivity without varying strength, durability, and permeability.

Effect of Exposure on Permeability of Panels:

Exposure had various effects on permeability. Of 117 panels, 14 remained unchanged in their permeability. These included nine panels which rated "excellent" (no dampness or leakage) and four which rated "good" (dampness only) throughout. Increase in age resulted in decreased permeability for 66 panels. Irregular results were obtained for 26 panels in that the 1-yr. test gave higher (or lower) permeabilities than were obtained at 35 days and 2½ yr. The ten panels that increased in permeability included two (14C2 and 19AW3) that failed by separation of brick and mortar before 1 yr. and three (11B2, 14B1, and 14B3) that failed before 2½ yr. One panel (15A3) failed before 35 days. It will be noted by reference to BMS60 (7) that bricks 11, 14, 15, and 19 had poor records in resisting 50 cycles of laboratory freezing and thawing. Improvement in permeability obviously will not result from exposure if disintegration is occurring.

In observing a long-time exposure test of brick-mortar assemblages, it was noted that the bricks in the top course of the wallettes had frequently become loosened after a winter's exposure. These same bricks after several wettings and dryings during the late spring and summer regained their bond. This "autogenous healing" (8) in combination with disintegration would seem a probable explanation of the behavior of the 26 panels in which the 1-yr. test showed greater (or less) permeability than was observed at both 35 days and 2½ yr. As with the exposure wallettes referred to above, this loss and recovery of bond appeared most frequently with mortars B and C, and was apparently a matter of seasonal variation. The same explanation may apply to large changes in permeability in one direction.

Dye-Permeability Tests:

Anderegg (4) used a dye solution applied with a brush to the edges of mortar joints. Such a procedure would indicate presence or absence of contact between brick and mortar at the edges of joints, but would not necessarily demonstrate the presence of through channels. A dye solution forced through a panel under a head of 1 in. of water is assumed to fill all through channels. The use of contrasting dyes (blue and yellow) with different times of contact serves to differentiate between coarse and fine channels. The larger channels appear yellow-green, smaller channels appear green to blue-green, and the smallest channels or capillaries are not filled by the short exposure to yellow dye and remain blue.

Limitations of space permit presentation of but three of the more than 100 diagrams of dye-permeability results obtained in this investigation. The results on panels 3A1, 3C3, and 11C1 are presented in Figs. 4, 5, and 6, as illustrations of typical cases. In these diagrams, the observer is looking down on the panel which is in the position it occupies during the permeability test. The brick-mortar interfaces are then separated along the lettered lines, as indicated. (This lettering does not indicate mortar types and the numbering of the individual bricks and half-bricks in the panel is unrelated to the brick-type identification numbers used in this paper.) The surfaces of these brick-mortar interfaces are then brought into view by rotating the individual bricks 90 deg. around their horizontal or vertical axes. In these diagrams, the absence of diagonal hatching indicates absence of stain. Closeness of hatching is proportional to intensity of stain, and the colors are distinguished by direction of hatching.

No dampness or leakage was observed for specimen 3A1, and these results agree with the dye-permeability diagram (Fig. 4). No penetration of dye was observed except for the "edge effects" on the face to which the dye was applied. These edge effects are the result of mortar being squeezed out of the joints when the bricks are pressed into place. (Such openings are especially noticeable on "struck" joints and have been mistaken by inexperienced observers for shrinkage cracks.) The dye was applied to the "tooled" face of the panel and the results indicate that "tooling" is effective in concealing these openings but, unless very carefully done, the cavity is not completely closed. The dye permeability results illustrated in Fig. 4 are typical for all of the panels which gave no dampness or leakage in the water permeability tests. In general, the least depth of edge penetration was noted on the panels constructed with mortar A. Mortar C gave relatively deep and irregular edge penetration.

The record on panel 3C3 was as follows: time for first damp, immediate; time for first drop of water, 1 min. 15 sec.; leakage at rate of 112 ml. per hour. It was noted by the observer of the 35-day test that "much of the leakage was through a crack in center brick," but Fig. 5 shows a through channel (dyed green) at the cut end of half-brick No. 5. Aside from the presence of the through channel, panel 3C3 differed from 3A1 only in having deeper edge penetration and exhibiting dyed edges on both faces of the specimen. This second difference results from spreading of the dye solution on the lower face of the panel after having leaked through interface 5-J and through the crack in brick 4.

The result on panel 3C3 is typical for specimens which leak through one or more through channels but whose mortar joints otherwise are impermeable. Depending upon the size and number of these through channels, the panels rate from "fair" to "very poor." If the channels are small (blue staining only), the permeability tests usually show dampness only (good). The through channels are usually at the ends of the bricks (vertical joints as laid), and the cut ends of the number 3 and 5 half-bricks are the most probable locations of leaks.

The results from the $2\frac{1}{2}$-yr. test on panel 11C1 were as follows: time for first dampness, immediate; time for first drop, immediate and flow at rate of 900 ml. per hour. The very considerable staining of the horizontal (bed) joints, as well as the vertical (end) joints, indicated in Fig. 6 will be noted. Figure 6 represents about the average appearance of dye-permeability tests on panels built from the soft-mud bricks Nos. 7, 9, 11, 14, and 15, laid dry. With these bricks of relatively high initial rate of absorption, no significant or characteristic differences in area or intensity of dye appeared to result from differences in the mortars. Increase in area and intensity was, however, related to increase in penetrability of the bricks. The observations on panels built with bricks 15 and 19 were uncertain, since considerable injury to the surfaces of these low-strength bricks occurred during removal of the mortar.

Dye-permeability tests on panels built from bricks 8, 9, 11, 14, 15, and 19, wetted and laid with mortar A, gave faint blue staining on the brick-mortar interfaces. This faint staining was, however, quite uniform and complete and may indicate transmission of dye through the bricks rather than along the brick-mortar interfaces. Except for mortar C, which was stained up to 1 in. back from the end of the edge penetration, the mortar did not appear to be permeable according to these results. These wetted bricks were very difficult to separate from the mortar and the difficulty increased with decrease in strength of the bricks. Brick 19, laid wet, was extreme in this respect. The best results from the use of two contrasting dyes for determining degree and nature of "bond" between brick and mortar were, in general, obtained on specimens made from bricks and mortar which are not themselves highly permeable to moisture.

Relation to Work of Previous Investigators:

Some of the bricks used in the present investigation correspond to certain brands and types used by Palmer and Parsons (**3**) and by Fishburn and his coworkers (**1**, **2**).

For the benefit of those interested, the tabulation of brick identities is given at the top of the next column.

Mortars A, B, and C of the present investigation correspond very closely in composition and properties to certain of the mortars used by Fishburn. Palmer's cement-lime-sand mortars (XTd, XRa, and XVb) correspond approximately in proportioning of materials to mortars A, B, and C, and, except for XTd which had a considerably lower water retentivity than mortar A, the properties are in reasonable agreement.

Brick Identification

Present paper	Fishburn		Palmer and Parsons (3)
	BMS7	BMS82	A.S.T M. (1934)
1	a	..	5
3[a]	..	A	2[a]
7	..	B	..
8	..	B	..
10	..	F	..
19	c	C	1

[a] Palmer No. 2 was formed by the end-cut process, whereas the present sample No. 3 was side-cut. Both samples were from the same plant and otherwise corresponded.

Fishburn used several different types of workmanship. His class A (BMS7 and 82) and class C (BMS7) are taken as the equivalent of the workmanship used in the present investigation, since complete filling of joints is the common feature. From Palmer's description of the technique used in constructing his test specimens, complete filling of joints should have been obtained.

All three investigators agree in finding that the best results were obtained with a brick of very low initial rate of absorption (No. 1 of this paper). They also agree in finding that improved results were obtained by wetting bricks having medium to high rates of absorption. Palmer's results with his mortar CII (1:3 lime-sand) and his comments on the permeability of his various mortars agree with the data of Fishburn and the present investigation, which indicate poorer results when high-lime mortars are used with impervious bricks. All three investigations provide data which indicate that, although permeability results are poor when bricks with high rates of absorption are used, irrespective of the mortar, results are somewhat improved by using mortars with increased water retentivity.

As would be expected in comparing results on panels $3\frac{3}{4}$ in. in thickness with panels 8 and 12 in. thick, the rates of flow per unit of time were very much greater in the present investigation than were those reported by Fishburn. Considering that Palmer's results were determined on specimens 8 in. in thickness and were reported as "average maximum rates" of three specimens, his rates of flow were extremely high. With the exception of his brick 5 and two tests on his brick 1, laid wet, all of Palmer's results rate "very poor" on Fishburn's and this scale.

The data of Fishburn and his coworkers (13) indicate, in general, that a definite and significant decrease in permeability resulted from the exposure of their specimens. The tendency of walls built from their brick C (No. 19 of the present paper) to undergo structural damage from exposure, accompanied in some instances by increase in permeability, was noted.

Summary and Conclusions

Small panels, two bricks long by three bricks high, and one brick ($3\frac{3}{4}$ in.) in thickness were built. Ten different kinds of brick were used. The average initial rates of absorption of these brick ranged from 0.5 to 127 g. per minute and the other properties such as absorption, porosity, air permeability, and strength also covered a considerable range. Three types of mortars corresponding to the A, B, and C mortars of the American Standard Building Code Requirements for Masonry were used. Good workmanship (all joints filled) was used throughout. In addition to the building of panels from these ten kinds of brick with three kinds of mortars, panels were built with the same kind of brick wetted before laying with mortar A.

All panels were tested at 35 days using the method described by Palmer and again tested after approximately 1-yr. exposure to weather, and again after $2\frac{1}{2}$-yr. exposure. Time for appearance of first dampness, first drop of water, and rate of flow in milliliters per hour was recorded on each test. The $2\frac{1}{2}$-yr. test was made with water solutions of blue and of yellow dyes used for periods of 2 hr. and 10 min. each respectively. Following this test, the panels were dissected and the extent, degree, and intensity of dye of the brick-mortar interfaces was observed and recorded.

Among the principal observations and conclusions are the following:

1. Under the conditions of test here used, only panels built from bricks with initial rates of absorption (penetrabilities) of less than 10 g. per minute and laid with mortar A, characteristically gave excellent results (no dampness or leakage up to 48 hr.) when tested. All panels built from bricks with higher initial rates of absorption (26 to 127 g. per minute) characteristically gave dampness or leakage (0 to over 5 liters per hour), irrespective of the mortar used. In general, the rate of leakage tended to increase with increase in initial rate of absorption of the bricks.

2. Other brick properties such as water absorption and porosity which in general increase with increase in penetrability, and strength, which is inversely related to penetrability, appear to be related to permeability of the panels, but the relation does not appear to be causal. Differences in air permeability and the derived "average radii of capillaries" and "number of capillaries per square centimeter" do not appear to be related to permeability within the ranges encountered in this work. Differences in textures of the brick surface in contact with mortar did

not appear to have much effect on permeability.

3. Prewetting of bricks with medium to high initial rates of absorption greatly improved results of the permeability tests in comparison with the same bricks laid dry and with mortar A used throughout. The permeability on panels laid with wetted bricks characteristically rated either "good" (dampness only) or "fair" (rate of flow less than 50 ml. per hour).

4. For the bricks and mortars used in this investigation, differences in bricks had much more effect on permeability than differences in the mortars.

5. Comparisons of permeability results on panels retested after 1 and 2½-yr. exposure, with initial tests made at 35 days, indicates, in general, that leakage decreased. Exceptions were provided by panels built from certain high-absorption and low-strength bricks which tended to disintegrate on exposure outdoors. The bricks which performed poorly had saturation coefficient exceeding 0.80.

6. By means of permeability tests using water solutions of dyes of contrasting colors applied for long and short times, the nature, extent, and location of through channels in permeable masonry specimens can be observed when the specimens are dissected. For the panels so examined, the most probable location of a single through channel is at the interface between the mortar and cut surface of a half-brick.

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(7) J. W. McBurney and J. C. Richmond, "Strength, Absorption and Resistance to Laboratory Freezing and Thawing of Building Brick Produced in the United States," BMS60, U. S. Govt. Printing Office, Nov. 5, 1940.

(8) Duff A. Abrams, "Tests of Bond Between Concrete and Steel", Univ. Illinois Eng. Exp. Sta., Bull. No. 71, p. 107, Dec. 1913.

(9) C. C. Fishburn, D. E. Parsons, and P. H. Petersen, "Effect of Outdoor Exposure on the Water Permeability of Masonry Walls," BMS76, U. S. Govt. Printing Office, Aug. 15, 1941.

DISCUSSION

MR. H. F. GONNERMAN[1] (*presented in written form*).—Mr. McBurney and his associates are to be commended for the good work they have done in studying the permeability of brick-mortar assemblages. The problem of damp or leaky walls is of the utmost concern to the construction industry and any studies that aid in the solution of the problem will be fully appreciated.

Although it was found in these studies that differences in bricks had more effect on permeability than differences in mortar, it seems appropriate to make some mention of the relatively good performance of mortar A. This mortar had the proportions corresponding to 1:0.25:3.9 by volume. Although this mortar had the lowest water retentivity of any of the three mortars used in these tests, it gave the highest strength and in comparison with the other two mortars generally gave good performance in the permeability tests. Previous studies have shown that mortar of this type gave high bond strength in brick-mortar assemblages.

The portland cement used in the preparation of the mortars was a type I portland cement. It seems probable that the use of an air-entraining cement would have produced an A mortar of higher water retentivity. Air entrainment tends to give "fatness" to mortars without increasing their permeability. At the same time the air entrainment is associated with only a moderate reduction in mortar strength.

If Mr. McBurney has plans for continuing these studies of the permeability of brick-mortar assemblages I should like to suggest that he include air-entraining portland cements in the preparation of the mortars. It is probable that the use of air-entraining cement would give even better results than those obtained with the type I cement used in the tests reported in the paper.

MR. F. O. ANDEREGG[2] (*presented in written form*).—Seventeen years ago we built over 300 panels at Mellon Institute in the study of leakage through brick walls.[3] Six of the panels were tight. One of these was built of bricks designated by the authors as brick A laid in cement mortar with $\frac{3}{8}$-in. thick joints.

In answer to Mr. Gonnerman's question:

The mortars in the other tight panels all contained ammonium stearate, an air-entraining agent, added to a typical 1:1:6 mix. The brick was less vitreous than brick A, having 1-min. absorption through one flat side of 10 to 20 g.

The panels were sprayed at intervals and the amount of leakage appeared to diminish with aging. The panels were exposed to the weather and rain water got in and apparently tended to promote some autogenous healing. Rain was drawn into all crevices by capillarity and carried in soot and dirt so that on demolishing the panels the paths of the leakage were readily discerned. Most flaws were between bricks and mortar, the paths having been there since the first laying. At first such crevices were not very apparent from outside, but as time went on and dirt collected therein they were readily discernible. I agree with the authors that when certain people talk about "shrinkage cracking" they are basing their estimate on incorrect observation.

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[1] Manager, Research Lab., Portland Cement Assn., Chicago, Ill.

[2] Consulting Specialist on Building Materials, John B. Pierce Foundation, Raritan, N. J.

[3] F. O. Anderegg, "Watertight Brick Masonry," *Architectural Record*, Vol. 70, p. 208 (1931).

Of our panels 98 per cent leaked. These were all erected by competent brick masons, showing the difficulty of getting the twenty odd inches periphery of each brick completely bonded to the mortar. It took us 3 months in the laboratory with the aid of the dye test to develop a technique to insure complete contact therebetween.[4] With very low-absorption bricks, a film of moisture tends to collect on the under side of the bricks, while where the absorption rate is high, the mortar congeals so fast that it usually fails to spread out over and make a continuous contact with the bricks.

MR. C. C. CONNOR[5] (*by letter*).—The most intriguing aspect of the results reported in this paper is the amazing resistance of 4-in. panels to penetration by a 1-in. head of water. It would seem that the "excellent" and "good" ratings, in general, reflect the penetrability of the brick and mortar materials themselves, laid up without any cracks whatever. If the results indicate the tendency of brick and mortar combinations to form complete bonds under field conditions they are valuable indeed, but if they only indicate what can be done in a laboratory, then the ratings are meaningless to the building industry. My data from studies of existing buildings and a rather wide field experience in construction indicate that these results do not give anything like a true picture of what would happen if these same combinations of brick and mortar were laid up in the field with full joints, concave tooling, push laid brick, and all of the other accepted requisites of excellent field workmanship.

The authors found that the differences in bricks had much more effect on the permeability of their panels than differences in the mortars. This may well have been true for their panels, but, if laboratory research is to serve as a guide for field practices, it is most unfortunate that the importance of mortars should have been minimized. The great majority of investigators on the subject of water penetration of brick walls agree that the properties of mortar are highly important. Evidence from the field certainly indicates the important influence of differences in mortars as well as in bricks.

The excellent results with bricks having very low rates of absorption reported in this paper are in definite disagreement with the indications of results obtained in the field. This is particularly true with bricks like No. 1 of the paper which had a maximum rate of absorption of 2 g. per minute, and averaged 0.5 g. It is likewise true for bricks like No. 3 which ranged from 1 to 23 g. per minute, and averaged 8 g., indicating that two or three of the six bricks in each panel were probably below 5 g. per minute. In field practice, such bricks would threaten the watertightness of a wall. In one building under my observation, test samples of the bricks averaged 3 g. in 1 min., and the mortar was a 1:1:5 mix with a high plasticity quicklime which produced excellent trowelability. Separation cracks developed within 24 hr. to such an extent that double tooling was necessary to prevent cracking and to obtain watertight walls. Areas where double tooling is known to have been omitted are characterized today by a high percentage of cracking between the brick and mortar. It can safely be assumed that this mortar had a water retentivity in excess of 75 per cent, and the same mortar with bricks having good rates of absorption between 5 and 25 g. produced low cracking and many watertight buildings without double tooling. In eleven buildings under my direct observation, where the bricks have very low rates of absorption—that is, below 5 g. in 1 min.—all except the one that was double-tooled have developed considerable separation cracking, and nine have leaked. Most of these buildings were built with low water-retentivity mortars, but another one was built with a 1:1:6 mortar mix with the lime being a finishing lime soaked for a 10-day period. This mortar should have had at least fair water retentivity. The building leaked, and the separation cracking was approximately 18 per cent as reported in my paper on separation cracking.[6]

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[4] F. O. Anderegg, "Some Properties of Mortars in Masonry," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 1130 (1940).

[5] Assistant Engineer, New Jersey Bell Telephone Co., Newark, N. J.

The discussion in this paper by McBurney, Copeland, and Brink indicates that the best results were obtained with a 1:3 plus 25 per cent of lime mortar mix and impervious bricks. Good results were obtained with the 1:1:5 mortar and impervious brick, but both of these mortars gave poorer results with bricks having higher rates of absorption. The 1:2:7.5 mix produced results that were not so good as the other mortars with the impervious bricks, but were better than the 1:3 plus 25 per cent of lime mortar with the highly absorptive bricks. The results of the highest lime content mortar, however, were erratic and, for the most part, were very poor to fair. In other words, it has been made quite plain that the effectiveness of the mortars decreased, in general, with their lime contents. This is a vastly different picture than that given by results obtained in the field. It is obvious that the low-lime, high-cement mortar is the most impervious of the three, and this would be very important when 4-in. brick panels are subjected to a 1-in. head of water if a complete bond was obtained between the brick and mortar. The difficulty in the field has been to get consistently a reasonably complete bond between high-cement mortars and the various kinds of brick. My observations on field construction indicate that the high-lime mortars like the 1:3 plus 50 per cent of lime and the 1:1:5, 1:1:6, and 1:2:7.5 mortars, regardless of water retentivity, consistently produce the lower percentages of cracking with bricks having rates of absorption that are neither very low nor high. They also produce a much higher percentage of watertight buildings than the low-lime, high-cement mortars. In the last seventeen years my company has built over 50 buildings with various high-lime mortars and bricks that had a low to moderate rate of absorption. All have remained watertight except three. This is much superior to any of the field results obtained with high-cement mortars.

The differences between the evidence given in this paper and the results in field practice are so great that the question arises as to how they could have occurred. It would appear that the reasons for the differences might well originate in the laboratory technique. I have been unable to observe field results with a high cement mortar truly comparable to mortar A because it is not in common use, but such results from mortars, similar except for lower water retentivities, are much different from those reported in this paper. In addition, buildings of impervious bricks and 1:1:5 mortars, like mortar B of the paper, can and have been observed, and the results reported in the paper for that combination of brick and mortar are so much superior to field results obtained with good workmanship, that the conclusion is inescapable that the laboratory methods used in some way produced unusual brickwork.

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[6] C. C. Connor, "Resultant Separation Cracking Between Various Mortars and Brick in Existing Brick Structures," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 454 (1934).

It is known that heavy pressure exerted on the brick when it is being laid will produce durable and complete bonds with high-cement mortars even though their water retentivities are very low. McBurney and his co-workers laid their panels with one end against a stop to prevent the lower bricks from being moved out of alignment when the upper ones were shoved into place. This would indicate considerable pressure was used.

It is also known that better results can be obtained when the mortar is not spread out over a number of bricks as is common in field practice. The panels reported on in this paper were only two stretcher bricks long and one brick in thickness, which made it impossible to spread the mortar over any great area. This in itself should produce results superior to those generally obtained in the field.

It should also be noted that the very impervious bricks used in the various studies of water penetration through panels and wallettes at the Bureau of Standards were thoroughly dry when laid. The authors of this paper and Fishburn dried their impervious bricks before laying. This dryness gave the surface of the bricks their maximum absorption, reduced or eliminated their tendency to "float," and produced a condition more favorable to complete bond than obtains in the field.

The curing method used by McBurney, Copeland, and Brink—27 days in the spray room and 7 days drying at 100 F.—was much different from exposure to weather, and was favorable to cement and most unfavorable to lime. This could have exerted an important influence.

The laboratory has the right to use any measures it sees fit to achieve results, but if those measures cannot be applied in the field they are valueless and the products are little more than laboratory curiosities.

It may be significant that the results reported in this paper and the results reported by Fishburn and his co-workers[7, 8] in preceding investigations are similar to each other but greatly superior to those obtained by Palmer and Parsons.[9] Palmer and Parsons worked with 8-in. wallettes, but, except for the wallettes with impervious bricks, their results did not approach the good results the authors of this paper achieved with 4-in. panels. The results of Palmer and Parsons were in turn superior to those ordinarily obtained with good workmanship in the field, especially with the combinations of high-cement mortar and impervious bricks. The differences between the two laboratory studies and between the laboratory and field results are in all probability due to the laboratory methods used.

It is strange that McBurney and his co-workers did not test any of the types of brick where all of the individual bricks had suction rates between 5 and 20 g. in 1 min. The indication that this was the best range for moistureproof walls was pointed out in 1934, and D. E. Parsons stated in 1939[10] that a much similar rate —0.2 to 0.7 oz.—had been indicated in tests to be the most desirable. In 4-in. panels such bricks would probably have shown dampness in the 24-hr. tests, but

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[7] C. C. Fishburn, D. Watstein, and D. E. Parsons, "Water Permeability of Masonry Walls," Nat. Bur. Standards Report BMS7.

[8] C. C. Fishburn, "Water Permeability of Walls Built of Masonry Units," Nat. Bur. Standards Report BMS82.

[9] L. A. Palmer and D. A. Parsons, "Permeability Tests of 8-in. Wallettes," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 419 (1934).

[10] D. E. Parsons, "Watertightness and Transverse Strength of Masonry Walls," Structural Clay Products Inst., October, 1939.

these bricks bleed slowly, if at all, and penetration through 12-in. walls would be very unlikely if the joints were watertight. In field construction these bricks not only have the advantage of consistently producing less cracking but also tend to absorb and retain the water that may enter through minor cracks. The apparent superiority of impervious face bricks and impervious mortar suddenly vanishes when a crack occurs because the water pours through unchecked to the back-up material, where the joints are seldom tight. One of the common problems of existing buildings—and one of the most difficult to solve—is the moistureproofing of walls of impervious face bricks laid up with high cement mortars which are leaking through a multitude of hair cracks.

The construction industry needs to know the combinations of brick and mortar and the types of workmanship which will consistently produce watertight walls, and the laboratory is the place to find the answers. But when the laboratory produces evidence that is contradictory in so many respects to extensive field experience and the data available from studies of existing buildings, it would seem the part of wisdom not to incorporate these laboratory finding into construction practice unless they are confirmed by field performance.

MR. J. W. MCBURNEY (*author's closure by letter*).—Mr. Gonnerman's suggestion that air-entraining cements be included in future work should certainly be followed. The most commonly used type of masonry cement is reported to be diluted portland cement with added plasticizing and air-entraining agents such as vinsol resin. Masonry cements now largely replace cement-lime mixtures.

Mr. Anderegg's work at Mellon Institute is recognized as one of the pioneer investigations in this field. It is doubtful, however, whether any of us in 1931 had much understanding of some of the elements of good workmanship that are now known to be the important ones from the standpoint of strength and impermeability of brick-mortar joints. Unless a mason is told what is wanted and is shown suitable techniques, the employment of "competent brick masons" is no guarantee of good workmanship as we now understand it. A prevalent idea at that time was that mortars for use with vitrified and semivitrified bricks should have a minimum of mixing water. The water-retentivity test for mortar had not been developed. Rate of absorption of brick was not widely recognized as an important factor.

Mr. Connor's objections to this paper appear to fall under the following heads:

1. The authors have minimized the importance of mortar;
2. "Excellent" results were obtained when mortar A was used with brick having penetrabilities of less than 10 g., which is contrary to Mr. Connor's experience;
3. "Excellent" and "good" results were obtained with other mortars on these same bricks which is also contrary to Mr. Connor's experience; and
4. Certain factors and variables were controlled which made the results better than they should be, in Mr. Connor's opinion.

It is somewhat surprising that Mr. Connor should ask for further demonstration of the effect of nonplastic, low-water-retentivity mortars on permeability. Have not Palmer and Parsons[9] covered that field? Their data, however, show most clearly that mortar is a minor factor compared with differences in brick. Mr. Connor's attention is called to their results on brick 5 with all ten of their mortars. Brick 5 is the brick 1 of the present paper. There is, incidentally, some evidence that the numerical values for permeabilities given by these investigators should have been in "milliliters per hour" rather than "milliliters per minute" as reported. The alternative of assuming that their workmanship was very defective is not in accord with their results on brick 5. If "milliliters per minute" is right, the absurdity of Mr. Connor's statement that "the results of Palmer and Parsons were in turn superior to those ordinarily obtained with good workmanship in the field—" is made evident by converting their results from milliliters per minute per ½ sq. ft. to gallons per hour per 1 sq. ft.

Mr. Connor apparently believes that the quality of a mortar is unalterably fixed by its proportions of cement to lime. There are others who believe the same. For example, one of the brick masons hired to construct panels as part of Mr. Fishburn's research had worked for several days using mortar A. (The general practice in this work was not to tell a mason what mortar he was using.) He was then supplied with a 1:2:7.5 mortar made with a mason's hydrate of such quality that the resulting water retentivity was about 40 per cent. The mason thereupon complained that the lime had been left out of the mortar! The authors prefer to judge mortars on the basis of their properties.

Mr. Connor reports that nine out of eleven buildings, built with brick of low rate of absorption, have leaked. On the grounds of the National Bureau of Standards there are seven major buildings constructed from brick 3. Not one of these buildings has ever leaked through its masonry according to the testimony of the Bureau's Superintendent of Maintenance. The clue to this difference may be found in the results of certain tests reported in BMS7 and 82. Using, for example, brick 1 (there called "a") and mortar A, panels 3 by 4 ft. were built by a commercial mason. Some of these panels gave no dampness or leakage with water under pressure for a period of several days. Other panels of the very same brick and mortar transmitted water in seconds under the same condition of test. The types of workmanship (A and B) that gave these different results are described in detail and illustrated. Also recommended is the booklet of the Louisville Cement Co., "Type of Workmanship Recommended to Secure Dry Brick Walls" which presents information on workmanship very simply and clearly and in more detail by pictures. Speaking frankly, Mr. Connor's results are as puzzling to the authors as theirs are to him. Washington has its share of speculative builders but they usually avoid jobs where they will be subjected to rigid inspection. Maybe quality of inspection is the answer. Practically all masonry looks alike when it is laid up and pointed. Unless an inspector who can use his eyes and who knows his business is present continually during construction, good workmanship is an unproved assumption.

This paper on permeability was a side issue of a much larger investigation on strength of bond of brick-mortar assemblages. It is hoped to publish this work in the near future. Some of Mr. Connor's criticism of laboratory procedures will be considered in that paper.

A METHOD OF PARTICLE SIZE DETERMINATION OF SOILS, CEMENT, ETC., BY MEANS OF A CHAINOMATIC SPECIFIC GRAVITY BALANCE*

BY EUGENE V. BARRETT[1]

SYNOPSIS

A method of particle size determination of soils, cements, and other fine-grained materials has been developed in the Materials Testing Laboratory of the Ministry of Public Works in Caracas, Venezuela. This method employs a chainomatic specific gravity balance with a 2-cu. cm. spherical glass plummet suspended by a corrosion-resistant steel wire 0.0008 in. in diameter. The method permits the accurate determination of the specific gravity of a suspension of fine particles in a liquid medium at any predetermined depth. The use of this method eliminates the computations and corrections of the hydrometer method and permits the determination of the percentage of soil particles finer than 0.0015 mm. to be made in 72 min. instead of the 24 hr. required by the hydrometer method. The determination of the specific surface of a sample of portland cement can be made in about 7½ min., particle size being determined to 0.005 mm. Check tests of soil samples indicate that this method is more accurate and has much better reproducibility than the hydrometer method. Check tests of cement samples indicate the specific gravity balance method is as accurate as the turbidimeter method.

The development of the method described in this paper was due to the dissatisfaction of the author with the hydrometer and turbidimeter methods of fine particle and specific surface determination. A study of Stokes' law indicated the advantages to be gained by developing a practical method of accurately determining the density of a suspension of fine particles at any predetermined level below the surface of the suspension.

The first experiments were made early in 1939 with a Westphal specific gravity balance employing a solid glass plummet with an over-all length of 8 cm. and with a displacement of 10 ml. This balance had a range of from 0.7000 to 2.0000 and could weigh to the fourth decimal place. The Westphal balance proved to be impractical for the rapid determination of the density of suspensions and the results obtained were not consistent. Furthermore the 8-cm. over-all length of the plummet did not permit the determination of the density of a suspension near the surface. Experiments were then begun with a chainomatic specific gravity balance employing a plummet with a displacement of 2 ml. These experiments were continued and a method has been developed which is practical and which permits the accurate determination of the particle size distribution of any fine-grained material dispersed in a suitable medium. This method is extremely rapid and eliminates the corrections

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

[1] Chief, Materials Testing Laboratory, Ministry of Public Works of Venezuela, Caracas, Venezuela.

and computations of the hydrometer and turbidimeter methods.

APPARATUS

The chainomatic specific gravity balance used with this method is a standard model made in the United States. The balance is made to operate with a glass plummet that displaces 2 ml. and the chain with vernier gives readings accurate to 0.0001 g. up to 0.1000 g. when the sliding weight is at the extreme right of the beam. For readings up to 2.0000 the sliding weight on the beam is moved to the left into the proper notches by means of a weight carrier. The balance is mounted on a glass base which has a hole about 3.5 in. in diameter with its center directly under the point of plummet suspension. The plummet furnished with the balance is not used in the procedure for particle size determination.

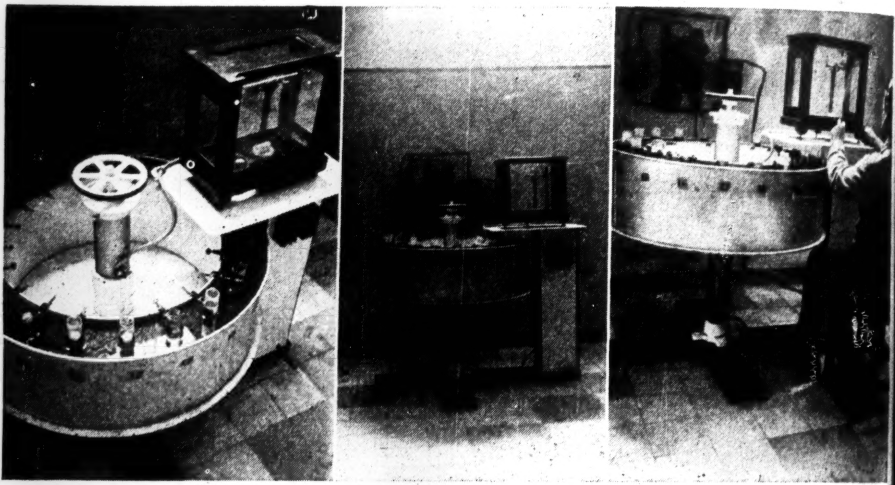

FIG. 1.—First Specific Gravity Balance Apparatus Constructed. Motor belt connected.

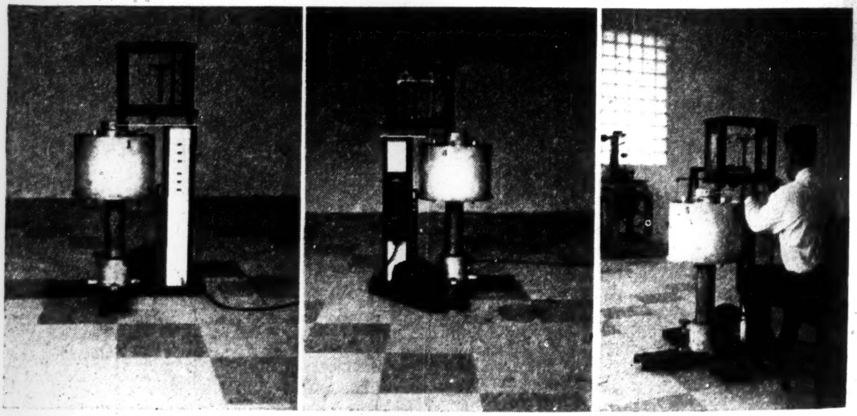

FIG. 2.—Front and Rear Views of Second Apparatus. Motor gear connected.

The balance is mounted on a horizontal steel plate (see Figs. 1 and 2) with a hole directly under the hole in the base of the balance and the position of the balance on the plate is exactly maintained by means of three shallow holes in which the legs of the balance rest. The horizontal steel plate is bolted to the top of a rectangular vertical column and the bottom of this column is bolted to the end of one of the legs of the base. The base of the apparatus consists of pieces of steel channel welded together as shown in Figs. 1 and 2 and a cylindrical steel column with diametrically opposite vertical slots is bolted to the base at the intersection of the channels. A steel screw is mounted inside the cylindrical column and a brass nut, with two legs which project out of the slots in the column, can be raised or lowered by revolving the screw. The legs of this nut support a cylindrical sleeve which slides up and down the machined surface of the cylindrical column, and this sleeve in turn is the central portion of a circular tank. The tank is free to revolve and is raised and lowered by means of a three-phase motor which is either belt or gear connected to the steel screw. The total vertical movement of the tank is 30 cm. and the time required to raise or lower the tank can be from 5 to 15 sec. The tank can be made to stop at any desired elevation by means of small switches suitably mounted. These small switches can be of such a type as shown in Fig. 3, mounted on the balance support column, or they can be a standard make of micro-switch mounted in one of the vertical slots of the cylindrical column. If micro-switches are used, the upper inside edge of the tank sleeve should be slightly beveled so that the beveled surface will push in the micro-switch bottoms the required distance to operate them, when the tank is raised. Since the tank sleeve can slide by the micro-switch bottoms, when they are in, the number of elevations at which the tank can be made to stop is limited only by the dimensions of the micro-switches. The small switches which stop the tank at the desired elevations operate a three-phase magnetic switch which is in turn con-

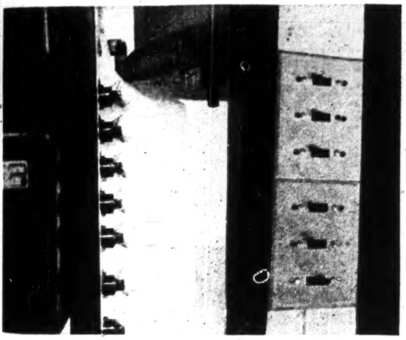

FIG. 3.—Small Switch Detail and Mounting of Double Throw Switches.

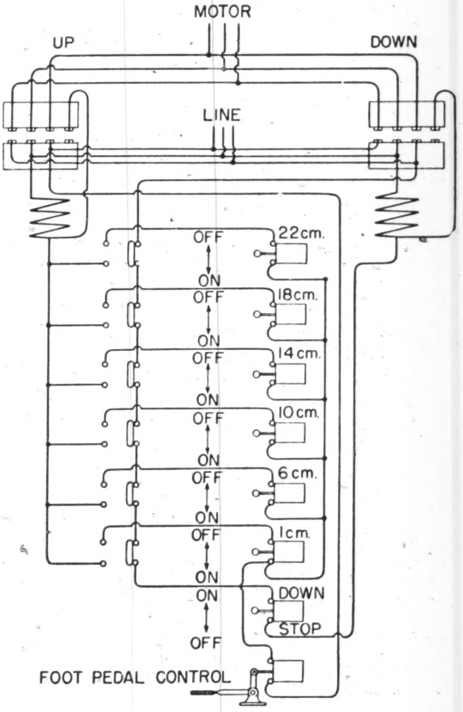

FIG. 4.—Wiring Diagram.

nected to the motor. Each small switch is connected to one side of a double throw switch mounted on the column supporting the balance (see Fig. 3). The other side of each of these double throw switches is connected to a three-phase magnetic switch which is in turn connected to the motor in such a manner as to result in lowering the tank when this circuit is closed. The wiring diagram for the small switches is shown in Fig. 4. To raise the tank to the required elevation it is only necessary to push the required switch to the opposite position. To lower the tank the same switch is returned to its original position. A lower limit switch, also shown in the wiring diagram, automatically stops the tank at the required lower elevation.

The tank has $\frac{1}{2}$-in. threaded holes in the bottom, so located that the center of each hole is exactly under the center of plummet suspension when the tank is revolved into the proper position. Each of these holes has a $\frac{1}{2}$-in. threaded brass rod screwed into it so that about one inch of the rod projects below the bottom of the tank. A lock nut is screwed onto the projecting lower end of each rod and a circular aluminum graduate support with a threaded hole in the center is screwed onto the upper end of each rod. These graduate supports have a beveled raised rim around the edge which centers the base of the graduates automatically and the supports can be raised or lowered either by means of the rods or by screwing or unscrewing the graduate supports on to the rods (see Fig. 5). The threads on the rods are purposely made oversize so that the graduate supports will remain in the positions in which they are placed.

The glass graduates are a standard make made in the United States and are graduated to 250 ml. The over-all height of the graduates is about 33 cm. and the inside height to the 250-ml. mark is about 26 cm. Graduates are selected which closely conform to these dimensions since a minimum of vertical adjustment is then required. Each graduate is numbered, the number is painted on the outside of the tank and the graduate holders are adjusted so as to have all of the 250-ml. marks on the graduates at the same elevation when they are revolved under the balance.

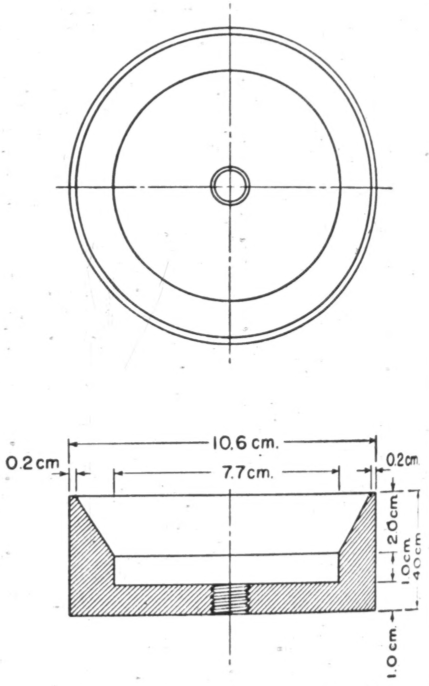

FIG. 5.—Graduate Support Details.

Each graduate is supported near its top by an adjustable snap clamp of the type that is used with Soxhlet extraction apparatus and these clamps are adjusted so that all of the graduates are vertical. The outer edge of the tank has a notch alongside of each graduate, and when the tank is revolved so that this notch engages a vertical guide, attached to the column supporting the balance, the center of the graduate is vertically under the suspended plummet and remains in this position as the tank is raised. The guide is pin connected at its upper end and the lower end is moved by a foot pedal so that a slot can be disengaged when the tank is in the down position but cannot be disengaged when the tank is up. An electric switch attached to the lower end of the guide only permits raising the tank when the guide is in the slot.

The balance plummet is a spherical glass ball approximately 1.56 cm. in diameter with the two ends of a piece of small-diameter platinum wire inserted into it so as to form a small flat projecting loop. The ball is made so that its original volume, including the platinum loop, is slightly in excess of 2 cu. cm. at 25 C. and is then ground, in such a manner that it remains spherical, until its volume is almost 2 cu. cm. at 25 C. It is then highly polished with rouge and polishing is continued until the chainomatic specific gravity balance has a reading of 0.9970 when the plummet is suspended in distilled water at 25 C. The plummet is suspended by a corrosion-resistant steel wire 0.0008 in. in diameter which is tied to the platinum loop of the plummet. This corrosion-resistant steel wire is the kind used in resistance strain gages and 22 cm. of this wire has a volume so small that the balance cannot appreciate it. The upper end of the wire is tied to a hook (see Fig. 6) and this hook is so made that it can be shortened by screwing the lower threaded portion into the upper part. The upper end of the hook is suspended from the chainomatic balance. The corrosion-resistant steel wire suspending the plummet is of such a length that the center of the glass plummet will be approximately 22 cm. below the 250-ml. mark on a graduate when the tank is in its highest position. The hook permits adjusting the height of the plummet so that its center will be exactly the required 22 cm. below the 250-ml. mark on the graduates.

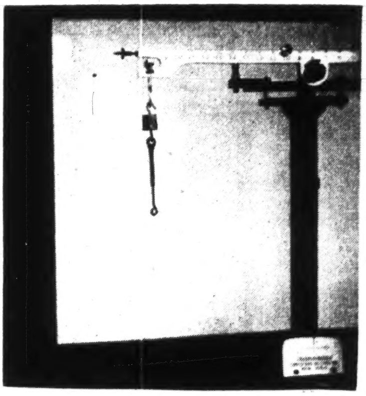

FIG. 6.—Details of Hook for Plummet Suspension.

TEST PROCEDURE FOR SOIL TESTING

Preparation of Sample:

The soil sample as received is oven dried at 110 C. and sieved through a No. 4 sieve. Any soil lumps remaining on the sieve are broken up and the minus No. 4 sieve material is passed through a sample splitting apparatus so as to obtain a representative sample of from 30 to 300 g., depending on the fineness of the soil. This sample is weighed and placed on a No. 200 sieve which is supported on a frame over a basin. The sample is washed with a pressure stream of water from a small hose and all of the wash water is retained in the basin. Washing is continued until the material retained on the sieve is clean. The basin is set aside and about 2 ml. of concentrated HCl is added to its contents. The HCl causes the soil to flocculate and it usually settles within a half hour. When the water above the soil is clear, the clear water is siphoned off and the basin is placed in a mechanical convection oven to dry at 110 C. When the soil is dry it is removed from the basin, broken up on a bread board with a rolling pin, and from 10 to 20 g. of the warm soil is placed in a desiccator to cool. The material retained on the No. 200 sieve is dried and its grading determined. When the minus No. 200 sieve material is cooled, 7 g. of it are weighed out for the particle size determination and 1 g. is weighed out to be used in the determination of the specific gravity of the soil.

Determination of Specific Gravity:

Small flasks are used in the determination of the specific gravity of the soils (see Fig. 7). These flasks were specially made and have an internal volume of about 14 ml. The approximate dimensions of these flasks are: over-all height 7 cm., height of bulb 4 cm., maximum external diameter of bulb 3 cm. and internal diameter of neck 0.6 cm. Each flask is made with a thick rim around the top of the neck, and glass is removed from these rims by grinding until all of the flasks have the same weight when suspended in distilled water at 25 C. The rims of the flasks are then heated until the surface that was ground fuses and becomes smooth. The flasks have two diametrically opposite holes in the neck immediately below the rim, and flasks can be readily suspended from a balance by inserting the hook, made of spring brass wire, shown in Fig. 7. The 1-g. samples for specific gravity determination are placed in the flasks, distilled water is added until the bulbs are about two-thirds full and the flasks are placed in a water bath with the necks above the surface of the water. The water bath is boiled for at least two hours, so as to eliminate any air that might have been trapped in the soil samples and the flasks and their contents are cooled to 25 C. The weight of each flask is then determined, while it is suspended in distilled water at 25 C. This weight is determined by means of a laboratory balance with the flask suspended in a beaker of water and with a tare weight, equal to the weight of the flask in water, on the right hand pan of the balance. The weight of one gram of the soil in water is therefore determined directly and the corresponding specific gravity of the soil is determined from Table I without any computation.

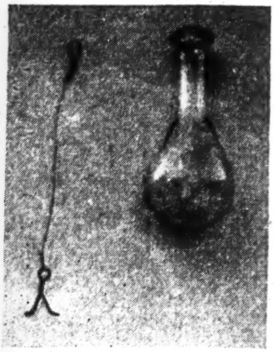

FIG. 7.—Small Flask for Determination of Specific Gravity of Soils and Hook for Suspending It from Balance.

TABLE I.—WEIGHT OF ONE GRAM OF SOIL IN WATER AND CORRESPONDING SPECIFIC GRAVITIES.

Weight of 1 g of Soil in Water	Specific Gravity of Soil	Weight of 1 g. of Soil in Water	Specific Gravity of Soil	Weight of 1 g. of Soil in Water	Specific Gravity of Soil
0.5868	2.42	0.6169	2.61	0.6429	2.80
0.5885	2.43	0.6183	2.62	0.6441	2.81
0.5902	2.44	0.6198	2.63	0.6454	2.82
0.5919	2.45	0.6212	2.64	0.6466	2.83
0.5935	2.46	0.6226	2.65	0.6479	2.84
0.5951	2.47	0.6241	2.66	0.6491	2.85
0.5968	2.48	0.6255	2.67	0.6503	2.86
0.5984	2.49	0.6269	2.68	0.6516	2.87
0.6000	2.50	0.6283	2.69	0.6528	2.88
0.6016	2.51	0.6296	2.70	0.6540	2.89
0.6032	2.52	0.6310	2.71	0.6552	2.90
0.6047	2.53	0.6324	2.72	0.6564	2.91
0.6063	2.54	0.6337	2.73	0.6575	2.92
0.6078	2.55	0.6350	2.74	0.6587	2.93
0.6094	2.56	0.6364	2.75	0.6599	2.94
0.6109	2.57	0.6377	2.76	0.6610	2.95
0.6124	2.58	0.6390	2.77	0.6622	2.96
0.6139	2.59	0.6403	2.78	0.6633	2.97
0.6154	2.60	0.6416	2.79	0.6644	2.98

Determination of the Specific Gravity of the Suspension:

The samples for particle size determination are soaked overnight in distilled water and dispersed with a standard dispersion apparatus (see A.S.T.M. Method of Mechanical Analysis of Soils (D 422 - 39).[2] Just prior to dispersion, 5 ml. of sodium silicate solution (density 1.020 at 25 C.), is added to each sample and the dispersed soil is washed into a 250-ml. graduate with sufficient distilled water to make 250 ml. of suspension. The water that is added is of such a temperature as to bring the suspension to 25 C. The water in the tank of the particle size apparatus is brought to 25 C. and the graduates are placed in position in the tank. Two minutes before the testing of a sample is to begin, the sample is taken out of the tank and dispersed for 1 min. with a motor-driven brush simular to but larger than the brush used with the Wagner turbidimeter apparatus (see Fig. 8). The sample is then dispersed by hand for the remaining minute and the graduate is placed in position in the tank. The graduate containing the sample is revolved into position under the balance and the tank is raised so that it will reach the required elevation not more than 7 sec. before a reading is to be taken. As soon as the reading is taken the tank is lowered, a glass Petri culture dish is held under the plummet and the plummet is washed with a stream of distilled water from a rubber bulb with a glass tube. We prefer to determine the specific gravity of a suspension at the times and with the values of L given below:

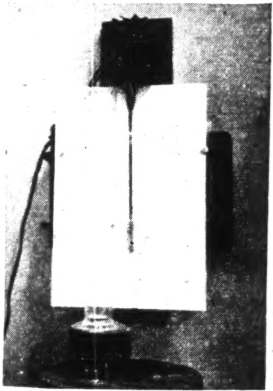

FIG. 8.—Brush Used for Dispersion of Soil Samples with 250-ml. Graduate Alongside.

$T = 2$ min. $L = 22$ cm.
$T = 9$ min. $L = 22$ cm.
$T = 18$ min. $L = 1$ cm.
$T = 64$ min. $L = 22$ cm.
$T = 72$ min. $L = 1$ cm.

Assumptions and Development of Formulas:

The fundamental assumptions relative to all sedimentation methods of particle size distribution, determination and the development of the several formulas used in this paper have been covered by a number of authors.[3]

Computations for Soil Testing:

Stokes' law gives the rate of fall of a small sphere in a liquid medium. If we solve Stokes' law for the diameter of the sphere we have:

$$d = \sqrt{\frac{30nL}{980(G - G_1)T}} \quad \ldots (1)$$

where:

d = the diameter of the sphere in millimeters,
n = the coefficient of viscosity of the medium in dyne-seconds per square centimeter,
L = the distance in centimeters through which the sphere has settled in time T,
G = the specific gravity of the sphere,
G_1 = the specific gravity of the liquid medium, and
T = the time in minutes for the sphere to settle a distance equal to L centimeters.

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[2] 1944 Book of A.S.T.M. Standards, Part II, p. 616.

[3] R. C. Thoreen, "Comments on the Hydrometer Method of Mechanical Analysis," *Public Roads*, Vol. 14, No. 6, August, 1933; and "Symposium on New Methods for Particle Size Determination in the Subsieve Range," Am. Soc. Testing Mats., March 4, 1941.

Since the values of L, n, G, and G_1 are known, it is possible to solve Stokes' law for d for the several values assigned to T. This has been done and the particle sizes for each specific gravity and for each value of T are given in Table II. In computing this table the following values were used:

L = as indicated in table,
n = 0.00894 which is the value for distilled water at 25 C.,
G = the specific gravity of the soil indicated in the table,
G_1 = 0.9975 which is the value for a mixture of distilled water and the indicated amount of sodium silicate, and
T = as indicated in the table.

Since 7 g. of oven-dry soil is always dispersed in sufficient distilled water plus sodium silicate to make 250 ml. of suspension, it follows that the weight of soil originally dispersed in 1 ml. of suspension will be a constant for all soils tested. The percentage of soil particles which have not settled below the plane at which the specific gravity of the suspension is determined can be computed

TABLE II.—PARTICLE SIZE IN MILLIMETERS, AT 25 C.

Specific Gravity of Soil	T = 2 min. L = 22 cm.	T = 9 min. L = 22 cm.	T = 18 min. L = 1 cm.	T = 64 min. L = 22 cm.	T = 72 min. L = 1 cm.
2.40	0.0463	0.0218	0.00329	0.00819	0.00165
2.42	0.0460	0.0216	0.00326	0.00813	0.00163
2.44	0.0456	0.0215	0.00324	0.00807	0.00162
2.46	0.0453	0.0213	0.00322	0.00802	0.00161
2.48	0.0450	0.0212	0.00320	0.00797	0.00160
2.50	0.0447	0.0211	0.00318	0.00791	0.00159
2.52	0.0444	0.0209	0.00315	0.00786	0.00158
2.54	0.0441	0.0208	0.00313	0.00781	0.00157
2.56	0.0439	0.0206	0.00311	0.00776	0.00156
2.58	0.0436	0.0205	0.00309	0.00771	0.00155
2.60	0.0433	0.0204	0.00307	0.00766	0.00154
2.62	0.0430	0.0203	0.00306	0.00761	0.00153
2.64	0.0428	0.0201	0.00304	0.00757	0.00152
2.66	0.0425	0.0200	0.00302	0.00752	0.00151
2.68	0.0422	0.0199	0.00300	0.00748	0.00150
2.70	0.0420	0.0198	0.00298	0.00743	0.00149
2.72	0.0418	0.0197	0.00297	0.00739	0.00148
2.74	0.0415	0.0195	0.00295	0.00735	0.00148
2.76	0.0413	0.0194	0.00293	0.00731	0.00147
2.78	0.0410	0.0193	0.00292	0.00726	0.00146
2.80	0.0408	0.0192	0.00290	0.00722	0.00145
2.82	0.0406	0.0191	0.00288	0.00718	0.00144
2.84	0.0404	0.0190	0.00287	0.00715	0.00144
2.86	0.0402	0.0189	0.00285	0.00711	0.00143
2.88	0.0399	0.0188	0.00284	0.00707	0.00142
2.90	0.0397	0.0187	0.00282	0.00703	0.00141
2.92	0.0395	0.0186	0.00281	0.00699	0.00141
2.94	0.0393	0.0185	0.00279	0.00696	0.00140

TABLE III.—PER CENT FINER ON BASIS OF OVEN-DRY WEIGHT OF SAMPLE.

Specific Gravity of Particles	Specific Gravity Balance Reading																			
	0.9975	0.9985	0.9995	1.0005	1.0015	1.0025	1.0035	1.0045	1.0055	1.0065	1.0075	1.0085	1.0095	1.0105	1.0115	1.0125	1.0135	1.0145	1.0155	Difference for 0.0001
2.40	0.0	6.1	12.2	18.3	24.4	30.6	36.7	42.8	48.9	55.0	61.1	67.2	73.3	79.4	85.6	91.7	97.8			0.611
2.42	0.0	6.1	12.2	18.2	24.3	30.4	36.5	42.5	48.6	54.7	60.8	66.8	72.9	79.0	85.1	91.1	97.2			0.608
2.44	0.0	6.0	12.1	18.1	24.2	30.2	36.2	42.3	48.3	54.4	60.4	66.5	72.5	78.5	84.6	90.6	96.7			0.604
2.46	0.0	6.0	12.0	18.0	24.0	30.0	36.0	42.1	48.1	54.1	60.1	66.1	72.1	78.1	84.1	90.1	96.1			0.601
2.48	0.0	6.0	11.9	17.9	23.9	29.9	35.8	41.8	47.8	53.8	59.7	65.7	71.7	77.7	83.6	89.6	95.6			0.597
2.50	0.0	5.9	11.9	17.8	23.8	29.7	35.7	41.6	47.5	53.5	59.4	65.4	71.3	77.3	83.2	89.1	95.1			0.594
2.52	0.0	5.9	11.8	17.7	23.6	29.6	35.5	41.4	47.3	53.2	59.1	65.0	70.9	76.8	82.8	88.7	94.6			0.591
2.54	0.0	5.9	11.8	17.6	23.5	29.4	35.3	41.2	47.0	52.9	58.8	64.7	70.6	76.5	82.3	88.2	94.1	100.0		0.588
2.56	0.0	5.9	11.7	17.6	23.4	29.3	35.1	41.0	46.8	52.7	58.5	64.4	70.2	76.1	81.9	87.8	93.6	99.5		0.585
2.58	0.0	5.8	11.6	17.5	23.3	29.1	34.9	40.8	46.6	52.4	58.2	64.0	69.9	75.7	81.5	87.3	93.2	99.0		0.582
2.60	0.0	5.8	11.6	17.4	23.2	29.0	34.8	40.6	46.4	52.2	57.9	63.7	69.5	75.3	81.1	86.9	92.7	98.5		0.579
2.62	0.0	5.8	11.5	17.3	23.1	28.8	34.6	40.4	46.1	51.9	57.7	63.4	69.2	75.0	80.7	86.5	92.3	98.0		0.576
2.64	0.0	5.7	11.5	17.2	23.0	28.7	34.4	40.2	45.9	51.7	57.4	63.1	68.9	74.6	80.4	86.1	91.8	97.6		0.574
2.66	0.0	5.7	11.4	17.1	22.9	28.6	34.3	40.0	45.7	51.4	57.1	62.9	68.6	74.3	80.0	85.7	91.4	97.1		0.571
2.68	0.0	5.7	11.4	17.1	22.8	28.4	34.1	39.8	45.5	51.2	56.9	62.6	68.3	74.0	79.6	85.3	91.0	96.7		0.568
2.70	0.0	5.7	11.3	17.0	22.7	28.3	34.0	39.6	45.3	51.0	56.6	62.3	68.0	73.6	79.3	85.0	90.6	96.3		0.566
2.72	0.0	5.6	11.3	16.9	22.6	28.2	33.8	39.5	45.1	50.8	56.4	62.0	67.7	73.3	79.0	84.6	90.2	95.9		0.563
2.74	0.0	5.6	11.2	16.8	22.5	28.1	33.7	39.3	44.9	50.5	56.2	61.8	67.4	73.0	78.6	84.2	89.9	95.5		0.561
2.76	0.0	5.6	11.2	16.8	22.4	28.0	33.6	39.1	44.7	50.3	55.9	61.5	67.1	72.7	78.3	83.9	89.5	95.1		0.559
2.78	0.0	5.6	11.1	16.7	22.3	27.9	33.4	39.0	44.6	50.1	55.7	61.3	66.8	72.4	78.0	83.6	89.1	94.7		0.557
2.80	0.0	5.5	11.1	16.6	22.2	27.7	33.3	38.8	44.4	49.9	55.5	61.0	66.6	72.1	77.7	83.2	88.8	94.3	99.9	0.554
2.82	0.0	5.5	11.1	16.6	22.1	27.6	33.2	38.7	44.2	49.7	55.3	60.8	66.3	71.8	77.4	82.9	88.4	93.9	99.5	0.552
2.84	0.0	5.5	11.0	16.5	22.0	27.5	33.0	38.5	44.0	49.5	55.0	60.6	66.1	71.6	77.1	82.6	88.1	93.6	99.1	0.550
2.86	0.0	5.5	11.0	16.5	21.9	27.4	32.9	38.4	43.9	49.4	54.8	60.3	65.8	71.3	76.8	82.3	87.7	93.2	98.7	0.548
2.88	0.0	5.5	10.9	16.4	21.9	27.3	32.8	38.2	43.7	49.2	54.6	60.1	65.6	71.0	76.5	82.0	87.4	92.9	98.3	0.546
2.90	0.0	5.4	10.9	16.3	21.8	27.2	32.7	38.1	43.6	49.0	54.4	59.9	65.3	70.8	76.2	81.7	87.1	92.5	98.0	0.544
2.92	0.0	5.4	10.8	16.3	21.7	27.1	32.5	38.0	43.4	48.8	54.2	59.7	65.1	70.5	75.9	81.4	86.8	92.2	97.6	0.542
2.94	0.0	5.4	10.8	16.2	21.6	27.0	32.4	37.8	43.2	48.6	54.1	59.5	64.9	70.3	75.7	81.1	86.5	91.9	97.3	0.541

from the specific gravity balance reading by means of the following formula:

$$P = \frac{100G(D - G_1)}{W(G - G_1)} \quad \ldots\ldots(2)$$

where:

G = the specific gravity of the soil particles,

D = the specific gravity balance reading,

G_1 = the specific gravity of the liquid medium = 0.9975,

W = the weight of soil originally dispersed in 1 ml. of suspension = 0.028 g.

P = the per cent of soil remaining in suspension at the plane at which the specific gravity of the suspension is determined.

Equation 2 has been solved for the several probable specific gravity balance readings and for the range of specific gravities of soils that will normally be encountered. The results are tabulated in Table III.

Before the test begins, the specific gravity of each soil and the particle sizes which will correspond to the several balance readings are recorded on the record sheet. As each specific gravity balance reading is determined, the percentage finer is immediately determined from Table III and the corresponding point is plotted on semi-log paper. The particle size distribution curve for each sample is in this way complete within one minute after the last balance reading, for each sample, is made. Table III has been expanded so as to include balance readings with intervals of 0.0002 and the expanded table has been printed and a copy mounted on two rolls in a small box with a transparent plastic top. The column "Specific Gravity of Particles" has been pasted on this top and the table is rotated by means of a knob which rotates a small gear attached to each roll thus causing the table to unroll from one roll as it is rolled into the other. Copies of this expanded table can be obtained without cost by writing to the author.

Specific Surface of Soils:

The surface area of all the particles of an average diameter d_a mm. in a gram of the original sample can be computed by the formula

$$S_{d_a} = \frac{0.6P_a}{Gd_a} \text{ sq. cm.} \quad \ldots\ldots(3)$$

where:

d_a = the average particle size,

P_a = the percentage of this average particle size in the sample, and

G = the specific gravity of the sample.

and the specific surface in square centimeters per gram of the soil sample is equal to the sum of the surface areas computed for each of the average particle sizes in the sample.

We have found it convenient to base our computations on the following particle sizes in millimeters:

	Average Particle Size, mm.
0.074 to 0.040 mm.	0.057
0.040 to 0.020 mm.	0.030
0.020 to 0.010 mm.	0.015
0.010 to 0.005 mm.	0.0075
0.005 to 0.0025 mm.	0.00375
0.0025 to 0.0015 mm.	0.0020
finer than 0.0015 mm.	0.0010

The percentage of each average particle size in a sample is obtained by scaling it from the particle size distribution curve that was plotted on semi-log paper while the test was in progress.

Equation 3 has been solved for the range of specific gravities ordinarily encountered. The surface areas corresponding to the several average particle sizes are given in Table IV. The author believes that considerable simplification would result from the classification of soils, relative to their particle size distribution, on the basis of their specific surface. Table IV has been expanded to include specific gravities with 0.02 intervals and per cent of particles

TABLE IV.—SURFACE AREA OF SOILS IN SQUARE CENTIMETERS FOR PARTICLES OF INDICATED AVERAGE DIAMETERS IN MILLIMETERS.

Per cent of Particles of Indicated Size in Sample	0.057	0.030	0.015	0.0075	0.00375	0.0020	0.0010	0.057	0.030	0.015	0.0075	0.00375	0.0020	0.0010	0.057	0.030	0.015	0.0075	0.00375	0.0020	0.0010
	SPECIFIC GRAVITY 2.40							SPECIFIC GRAVITY 2.45							SPECIFIC GRAVITY 2.50						
1	4.40	8.34	16.66	33.34	66.66	125.00	250.00	4.30	8.16	16.34	32.66	65.34	122.50	245.00	4.22	8.00	16.00	32.00	64.00	120.00	240.00
2	9	17	33	67	133	250	500	9	16	33	65	131	245	490	8	16	32	64	128	240	480
4	18	33	67	133	267	500	1 000	17	33	65	131	261	490	980	17	32	64	128	256	480	960
6	26	50	100	200	400	750	1 500	26	49	98	196	392	735	1 470	25	48	96	192	384	720	1 440
8	35	67	133	267	533	1000	2 000	34	65	131	261	523	980	1 960	34	64	128	256	512	960	1 920
10	44	83	167	333	667	1250	2 500	43	82	163	327	653	1225	2 450	42	80	160	320	640	1200	2 400
12	53	100	200	400	800	1500	3 000	52	98	196	392	784	1470	2 940	51	96	192	384	768	1440	2 880
14	62	117	233	467	933	1750	3 500	60	114	229	457	915	1715	3 430	59	112	224	448	896	1680	3 360
16	70	133	267	533	1067	2000	4 000	69	131	261	523	1045	1960	3 920	67	128	256	512	1024	1920	3 840
18	79	150	300	600	1200	2250	4 500	77	147	294	588	1176	2205	4 410	76	144	288	576	1152	2160	4 320
20	88	167	333	667	1333	2500	5 000	86	163	327	653	1307	2450	4 900	84	160	320	640	1280	2400	4 800
22	97	183	367	733	1467	2750	5 500	95	180	359	719	1437	2695	5 390	93	176	352	704	1408	2640	5 280
24	105	200	400	800	1600	3000	6 000	103	196	392	784	1578	2940	5 880	101	192	384	768	1536	2880	5 760
26	114	217	433	867	1733	3250	6 500	112	212	425	849	1699	3185	6 370	110	208	416	832	1664	3120	6 240
28	123	233	467	933	1867	3500	7 000	120	229	457	915	1829	3430	6 860	118	224	448	896	1792	3360	6 720
30	132	250	500	1000	2000	3750	7 500	129	245	490	978	1960	3675	7 350	126	240	480	960	1920	3600	7 200
32	141	267	533	1067	2133	4000	8 000	138	261	523	1045	2091	3920	7 840	135	256	512	1024	2048	3840	7 680
34	148	283	567	1133	2267	4250	8 500	146	278	555	1111	2221	4165	8 330	143	272	544	1088	2176	4080	8 160
36	158	300	600	1200	2400	4500	9 000	155	294	588	1176	2352	4410	8 820	152	288	576	1152	2304	4320	8 640
38	167	317	633	1267	2533	4750	9 500	163	310	621	1241	2483	4655	9 310	160	304	608	1216	2432	4560	9 120
40	176	333	667	1333	2667	5000	10 000	172	327	653	1306	2613	4900	9 800	168	320	640	1280	2560	4800	9 600
42	184	350	700	1400	2800	5250	10 500	181	343	686	1372	2744	5145	10 290	177	336	672	1344	2688	5040	10 080
44	193	367	733	1467	2933	5500	11 000	189	359	719	1437	2875	5390	10 780	185	352	704	1408	2816	5280	10 560
46	202	383	767	1533	3067	5750	11 500	198	376	751	1503	3005	5635	11 270	194	368	736	1472	2944	5520	11 040
48	211	400	800	1600	3200	6000	12 000	206	392	784	1568	3136	5880	11 760	202	384	768	1536	3072	5760	11 520
50	220	417	833	1667	3333	6250	12 500	215	408	817	1633	3267	6125	12 250	211	400	800	1600	3200	6000	12 000
	SPECIFIC GRAVITY 2.55							SPECIFIC GRAVITY 2.60							SPECIFIC GRAVITY 2.65						
1	4.12	7.84	15.66	31.34	62.66	117.50	235.00	4.06	7.70	15.40	30.80	61.60	115.50	231.00	3.96	7.54	15.08	30.14	60.26	113.00	226.00
2	8	16	31	63	125	235	470	8	15	31	62	123	231	462	8	15	30	60	121	226	452
4	17	31	63	125	251	470	940	16	31	62	123	246	462	924	16	30	60	121	241	452	904
6	25	47	94	188	376	705	1 410	24	46	92	185	370	693	1 386	24	45	90	181	362	678	1 356
8	33	63	125	251	501	940	1 880	32	62	123	246	493	924	1 848	32	60	121	241	482	904	1 808
10	41	78	157	313	627	1175	2 350	41	77	154	308	616	1155	2 310	40	75	151	301	603	1130	2 260
12	50	94	188	376	752	1410	2 820	49	92	185	370	739	1386	2 772	48	90	181	362	723	1356	2 712
14	58	110	219	439	877	1645	3 290	57	108	216	431	862	1617	3 234	56	106	211	422	844	1582	3 164
16	66	125	251	501	1003	1880	3 760	65	123	246	493	986	1848	3 696	63	121	241	482	964	1808	3 616
18	74	141	282	564	1128	2115	4 230	73	139	277	554	1109	2079	4 158	71	136	271	542	1085	2034	4 068
20	83	157	313	627	1253	2350	4 700	81	154	308	616	1232	2310	4 620	79	151	301	603	1205	2260	4 520
22	91	172	345	689	1379	2585	5 170	89	169	339	678	1355	2541	5 082	87	166	332	663	1326	2486	4 972
24	99	188	376	752	1504	2820	5 640	97	185	370	739	1478	2772	5 544	95	181	362	723	1446	2712	5 424
26	107	204	407	815	1629	3055	6 110	105	200	400	801	1602	3003	6 006	103	196	392	784	1567	2938	5 876
28	115	219	439	877	1755	3290	6 580	114	216	431	862	1725	3234	6 468	111	211	422	844	1688	3164	6 328
30	124	235	470	940	1880	3525	7 050	122	231	462	924	1848	3465	6 930	119	226	452	904	1808	3390	6 780
32	131	251	501	1003	2005	3760	7 520	130	246	493	986	1971	3696	7 392	127	241	482	964	1929	3616	7 232
34	140	266	533	1065	2131	3995	7 990	138	262	524	1047	2094	3927	7 854	135	256	512	1025	2049	3842	7 684
36	148	282	564	1128	2256	4230	8 460	146	277	554	1109	2218	4158	8 316	143	271	542	1085	2170	4068	8 136
38	157	298	595	1191	2381	4465	8 930	154	293	585	1170	2341	4389	8 778	151	286	573	1145	2290	4294	8 588
40	165	313	627	1253	2507	4700	9 400	162	308	616	1232	2464	4620	9 240	159	301	603	1205	2411	4520	9 040
42	173	329	658	1316	2632	4935	9 870	170	323	647	1294	2587	4851	9 702	167	316	633	1266	2531	4746	9 492
44	181	345	689	1378	2757	5170	10 340	178	339	678	1355	2710	5082	10 164	175	332	663	1326	2652	4972	9 944
46	190	360	721	1441	2883	5405	10 810	186	354	708	1417	2834	5313	10 626	182	347	693	1386	2772	5198	10 396
48	198	376	752	1504	3008	5640	11 280	195	370	739	1478	2957	5544	11 088	190	362	723	1446	2893	5424	10 848
50	206	392	783	1567	3133	5875	11 750	203	385	770	1540	3080	5775	11 550	198	377	754	1507	3013	5650	11 300

Specific Gravity 2.70

1	3.90	7.40	14.80	29.60	59.20	111.00	222.00
2	8	15	30	59	118	222	444
4	16	30	59	118	237	444	888
6	23	44	89	178	356	666	1 332
8	31	59	118	237	474	888	1 776
10	39	74	148	296	592	1110	2 220
12	47	89	178	355	710	1332	2 664
14	55	104	207	414	829	1554	3 108
16	62	118	237	474	947	1776	3 552
18	70	133	266	533	1066	1998	3 996
20	78	148	296	592	1184	2220	4 440
22	86	163	326	651	1302	2442	4 884
24	94	178	355	710	1421	2664	5 328
26	101	192	385	770	1539	2886	5 772
28	109	207	414	829	1658	3108	6 216
30	117	222	444	888	1776	3330	6 660
32	125	237	474	947	1894	3552	7 104
34	132	252	503	1006	2013	3774	7 548
36	140	266	533	1065	2131	3996	7 992
38	148	281	552	1125	2250	4218	8 436
40	156	296	592	1184	2368	4440	8 880
42	164	311	622	1243	2486	4662	9 324
44	171	326	651	1302	2605	4884	9 768
46	179	340	681	1362	2723	5106	10 212
48	187	355	710	1421	2842	5328	10 656
50	195	370	740	1480	2960	5550	11 100

Specific Gravity 2.75

1	3.82	7.26	14.54	29.06	58.14	109.00	218.00
2	8	15	29	58	116	218	436
4	15	29	58	116	233	436	872
6	23	44	87	164	349	654	1 308
8	31	58	116	233	465	872	1 744
10	38	73	145	291	581	1090	2 180
12	46	87	174	349	698	1308	2 616
14	54	102	204	407	814	1526	3 052
16	61	116	233	465	930	1744	3 488
18	69	131	262	523	1046	1962	3 924
20	77	145	291	581	1163	2180	4 360
22	84	160	320	640	1279	2398	4 796
24	92	174	349	698	1395	2616	5 232
26	100	189	378	756	1512	2834	5 668
28	107	204	407	814	1628	3052	6 104
30	115	218	436	872	1744	3270	6 540
32	122	233	465	930	1860	3488	6 976
34	130	247	494	988	1976	3706	7 412
36	138	262	523	1046	2093	3924	7 448
38	145	276	552	1105	2209	4142	8 284
40	153	291	581	1163	2325	4360	8 720
42	161	305	610	1221	2442	4578	9 156
44	168	320	640	1279	2558	4796	9 592
46	176	334	669	1337	2674	5014	10 028
48	184	349	698	1395	2790	5232	10 464
50	191	363	727	1453	2907	5450	10 900

Specific Gravity 2.80

1	3.76	7.14	14.26	28.54	57.06	107.00	214.00
2	8	14	29	57	114	214	428
4	15	29	57	114	228	428	856
6	23	43	86	171	342	642	1 284
8	30	57	114	228	457	856	1 712
10	38	71	143	285	571	1070	2 140
12	45	86	171	342	685	1284	2 568
14	53	100	200	400	799	1498	2 996
16	60	114	228	457	913	1712	3 424
18	68	128	257	514	1027	1926	3 852
20	75	143	285	571	1141	2140	4 280
22	83	157	314	628	1256	2354	4 708
24	90	171	342	685	1370	2568	5 136
26	98	186	371	742	1484	2782	5 564
28	105	200	400	799	1598	2996	5 992
30	113	214	428	856	1712	3210	6 420
32	120	228	457	913	1826	3424	6 848
34	128	243	485	970	1940	3638	7 276
36	135	257	514	1027	2054	3852	7 704
38	143	271	542	1084	2169	4066	8 132
40	150	285	571	1141	2283	4280	8 560
42	158	300	599	1198	2397	4494	8 988
44	165	314	628	1256	2511	4708	9 416
46	173	328	656	1313	2625	4922	9 844
48	180	342	685	1370	2739	5136	10 272
50	188	357	713	1427	2853	5350	10 700

Specific Gravity 2.85

1	3.70	7.04	14.06	28.14	56.26	105.50	211.00
2	7	14	28	56	113	211	422
4	15	28	56	113	225	422	844
6	22	42	84	169	338	633	1 266
8	30	56	113	225	450	844	1 688
10	37	70	141	281	563	1055	2 110
12	44	84	169	338	675	1266	2 532
14	52	99	197	394	788	1477	2 954
16	59	113	225	450	900	1688	3 376
18	67	127	253	506	1013	1899	3 798
20	74	141	281	563	1125	2110	4 220
22	81	155	310	619	1238	2321	4 642
24	89	169	338	675	1350	2532	5 064
26	96	183	366	732	1463	2743	5 486
28	104	197	394	788	1576	2954	5 908
30	111	211	422	844	1688	3165	6 330
32	119	225	450	900	1801	3376	6 752
34	126	239	478	957	1913	3587	7 174
36	133	253	506	1013	2026	3798	7 596
38	141	267	535	1069	2138	4009	8 018
40	148	281	563	1125	2251	4220	8 440
42	156	295	591	1182	2363	4431	8 862
44	163	310	619	1238	2476	4642	9 284
46	170	324	647	1294	2588	4853	9 706
48	178	338	675	1350	2701	5064	10 128
50	185	352	703	1407	2813	5275	10 550

Specific Gravity 2.90

1	3.64	6.90	13.80	27.60	55.20	103.50	207.00
2	7	14	28	55	110	207	414
4	15	28	55	110	221	414	828
6	22	41	83	166	331	621	1 242
8	29	55	110	221	442	828	1 656
10	36	69	138	276	552	1035	2 070
12	44	83	166	331	662	1242	2 484
14	51	97	193	386	773	1449	2 898
16	58	110	221	442	883	1656	3 312
18	65	124	248	497	994	1863	3 726
20	73	138	276	552	1104	2070	4 140
22	80	152	304	607	1214	2277	4 554
24	87	166	331	662	1325	2484	4 968
26	94	179	359	718	1435	2691	5 382
28	102	193	386	773	1546	2898	5 796
30	109	207	414	828	1656	3505	6 210
32	116	221	442	883	1766	3312	6 624
34	124	235	469	938	1877	3519	7 038
36	131	248	497	994	1987	3726	7 452
38	138	262	524	1049	2098	3933	7 866
40	145	276	552	1104	2208	4140	8 280
42	153	290	580	1159	2318	4347	8 694
44	160	304	607	1214	2429	4554	9 108
46	167	317	635	1270	2539	4761	9 522
48	174	331	662	1325	2650	4968	9 936
50	182	345	690	1380	2760	5175	10 350

Specific Gravity 2.95

1	3.56	6.76	13.54	27.06	54.14	101.50	203.00
2	7	14	27	54	108	203	406
4	14	27	54	108	217	406	812
6	21	41	81	162	325	609	1 218
8	29	54	108	217	433	812	1 624
10	36	68	135	271	541	1015	2 030
12	43	81	162	325	650	1218	2 436
14	50	95	190	379	758	1421	2 842
16	57	108	217	433	866	1624	3 248
18	64	122	244	487	974	1827	3 654
20	71	135	271	541	1083	2030	4 060
22	78	149	298	596	1191	2233	4 466
24	86	162	325	650	1299	2436	4 872
26	93	176	352	704	1408	2639	5 278
28	100	190	379	758	1516	2842	5 684
30	107	203	406	812	1624	3045	6 090
32	114	217	433	866	1732	3248	6 496
34	121	230	460	920	1841	3451	6 902
36	128	244	487	974	1949	3654	7 308
38	135	257	514	1029	2057	3857	7 714
40	143	271	541	1083	2165	4060	8 120
42	150	284	568	1137	2274	4263	8 526
44	157	298	596	1191	2382	4466	8 932
46	164	311	623	1245	2490	4669	9 338
48	171	325	650	1299	2598	4872	9 744
50	178	338	677	1353	2707	5075	10 150

from 0.1 to 1.0 per cent by tenths and from 1 to 50 per cent with 1 per cent intervals. This expanded table has been printed and a copy has been mounted in a box similar to, but larger than, the box used in mounting Table III. Copies of this expanded table can be obtained, without cost, by writing to the author.

Test Procedure for Determining the Specific Surface of Portland Cement

Determination of Specific Gravity and Viscosity of Kerosine:

The kerosine used in determining the specific surface of portland cement is purchased in quantity and redistilled before it is used. The specific gravity of the kerosine is determined at 25 C. with the specific gravity balance and the calibrated plummet. The viscosity of the kerosine is determined by surrounding the Wagner turbidimeter timing burette with a bath at 25 C. and filling the burette with kerosine at 25 C. The time in minutes required for the surface of the kerosine to arrive at the 7.5 mark on the burette is recorded, and since this is the time required for a 0.0075-mm. particle to settle 2.1 cm. in kerosine of the viscosity and density of the sample,[4] it is possible to solve Stokes' law for n since

$$n = \frac{980\, d^2(G - G_1)T}{30L} \quad \text{.....} (4)$$

where:

$d = 0.0075$ mm.,
$G = 3.15$,
$G_1 =$ the specific gravity of the kerosine,
$T =$ the time in minutes, and
$L = 2.1$ cm.

Dispersion of Sample:

A 7-g. sample of oven-dry cement is placed in a test tube with kerosine and 4 drops of oleic acid are added. The sample is then dispersed for 1 min. with the brush dispersion apparatus used with the Wagner turbidimeter. The contents of the test tube are washed with kerosine, at 25 C., into a graduate and kerosine is added until the surface is at the 250-ml. mark. The suspension is then hand-dispersed for 1 min. after which the graduate is placed in position in the tank.

Determination of Specific Gravity of Suspension:

The times at which the specific gravity of the suspension is determined is dependent on the specific gravity and viscosity of the lot of kerosine. Since according to Stokes' law

$$T = \frac{30nL}{980\, d^2(G - G_1)} \quad \text{.....} (5)$$

it is possible to fix the value of the several particle sizes to be determined and the value of L for each determination and solve for T the time at which the specific gravity of the suspension should be determined. We have, up to the present time, used the following values of L and d:

$L = 22$ cm. $d = 0.055$ mm.
$L = 1$ cm. $d = 0.010$ mm.
$L = 22$ cm. $d = 0.040$ mm.
$L = 1$ cm. $d = 0.0075$ mm.
$L = 22$ cm. $d = 0.025$ mm.
$L = 1$ cm. $d = 0.005$ mm.

The corresponding values of T for the lot of kerosine we are using (specific gravity 0.8033 and viscosity 0.0145) are 1 min. 23 sec., 1 min. 53 sec., 2 min. 36 sec., 3 min. 22 sec., 6 min. 40 sec., and 7 min. 35 sec. The test procedure is the same as for soils with the exception that most of the foam that forms on the surface of some samples, due to hand dispersion, is removed before the plummet is introduced into the graduate. The plummet should be always thor-

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[4] Method of Test for Fineness of Portland Cement by the Turbidimeter. 1944 Book of A.S.T.M. Standards, Part II, p. 1214, see Table I.

TABLE V.—SURFACE AREA OF PORTLAND CEMENT IN SQUARE CENTIMETERS FOR PARTICLES OF INDICATED AVERAGE DIAMETERS.

Average Particle Size, mm.	Per Cent of Particles of Indicated Size in Original Sample																									Surface Area for 1 per cent, sq. cm.
	2	4	6	8	10	12	14	16	18	20	22	24	26	28	30	32	34	36	38	40	42	44	46	48	50	
0.0890	4.3	8.6	12.8	17.1	21.4	25.7	30.0	34.2	38.5	42.8	47.1	51.4	55.6	59.9	64.2	68.5	72.8	77.0	81.3	85.6	89.9	94.2	98.4	102.7	107.0	2.14
0.0645	5.9	11.8	17.7	23.6	29.5	35.4	41.3	47.2	53.2	59.1	65.0	70.9	76.8	82.7	88.6	94.5	100.4	106.3	112.2	118.1	124.0	129.9	135.8	141.7	147.7	2.95
0.0475	8.0	16.0	24.1	32.1	40.1	48.1	56.1	64.2	72.2	80.2	88.2	96.2	104.3	112.3	120.3	128.3	136.3	144.4	152.4	160.4	168.4	176.4	184.5	192.5	200.5	4.01
0.0325	11.7	23.4	35.2	46.9	58.6	70.3	82.1	93.8	105.5	117.2	128.9	140.7	152.4	164.1	175.8	187.5	199.3	211.0	222.7	234.4	246.1	257.9	269.6	281.3	293.0	5.86
0.0175	21.8	43.5	65.3	87.1	108.8	130.6	152.4	174.1	195.9	217.7	239.5	261.2	283.0	304.8	326.5	348.3	370.1	391.8	413.6	435.4	457.1	458.9	500.7	522.4	544.2	10.88
0.00875	43.5	87.1	130.6	174.1	217.7	261.2	304.8	348.3	391.8	435.4	478.9	522.4	566.0	609.5	653.1	696.6	740.1	783.7	827.2	870.7	914.3	957.8	1001.4	1044.9	1088.4	21.77
0.00625	61.0	121.9	182.9	243.8	304.8	365.7	426.7	487.6	548.6	609.5	670.5	731.4	792.4	853.3	914.3	975.2	1036.2	1097.1	1158.1	1219.0	1280.0	1341.0	1401.9	1462.9	1523.8	30.48
0.00350	108.8	217.7	326.5	435.4	544.2	653.1	761.9	870.7	979.6	1088.4	1197.3	1306.1	1415.0	1523.8	1632.7	1741.5	1850.3	1959.2	2068.0	2176.9	2285.7	2394.6	2508.4	2612.2	2721.1	54.42

NOTE.—Surface area for all particles indicated = specific surface in sq. cm. per gram of original sample.

oughly cleaned with CCl_4 before and after it is used in kerosine.

Computations:

A table containing the percentage finer for the several possible specific gravity balance readings and for the specific gravity of cement of 3.15 is computed for each lot of kerosine. This table is computed in the same way that Table III was computed and is similar to it. The percentage finer is then taken from the table for each balance reading and indicated particle size.

The percentage of cement retained on a No. 200 sieve is determined by dry sieving and the average particle sizes computed on the basis of particle fractions with the following diameters. (The plus 200 sieve material is assumed to have an average particle size between the Nos. 150 and 200 sieves).

	Average Particle Size, mm.
0.104 to 0.074 mm.	0.0890
0.074 to 0.055 mm.	0.0645
0.055 to 0.040 mm.	0.0475
0.040 to 0.025 mm.	0.0325
0.025 to 0.010 mm.	0.0175
0.010 to 0.0075 mm.	0.00875
0.0075 to 0.0050 mm.	0.00625
0.0050 to 0.0000 mm.	0.00350

The surface areas for the range of possible percentages of the several average particle sizes have been computed and are given in Table V. The values were computed from Eq. 3, using a specific gravity of cement equal to 3.15. For any test the surface area of the sample in square centimeters per gram is equal to the sum of the surface areas for all of the particles indicated.

TEST RESULTS

Particle Size of Soils:

In order to determine the reproducibility of test results obtained with the chainomatic specific gravity balance method of particle size determination, 14 soil samples were prepared and 10 consecutive determinations of the particle size distribution of each sample were made with the hydraulically operated apparatus. Since five specific gravity balance readings were required for each test of each sample, the total number of readings was 700. The average balance reading for each sample for each time interval was then computed and on the basis of these average readings it was found that:

6.4 per cent of the percentages finer had a variation of 0.0 per cent
44.2 per cent of the percentages finer had a variation within 0.3 per cent
73.2 per cent of the percentages finer had a variation within 0.6 per cent
89.2 per cent of the percentages finer had a variation within 0.9 per cent
97.7 per cent of the percentages finer had a variation within 1.2 per cent
99.4 per cent of the percentages finer had a variation within 1.5 per cent

This reproducibility is better than can be obtained with any other method with the possible exception of the time-consuming pipette method of the U. S. Bureau of Chemistry and Soils.

The results obtained by means of the specific gravity balance method and by the hydrometer method[2] were compared by testing eight duplicate samples of soils by the two methods. The hydrometer tests were made using 28 g. of the minus No. 200 sieve material that had been prepared for the specific gravity balance method and sodium silicate solution was added in the same proportion for all of these tests. The results obtained were, for comparative purposes, converted to the basis of specific surfaces and are given below:

COMPARATIVE RESULTS OBTAINED BY MEANS OF THE SPECIFIC GRAVITY BALANCE AND HYDROMETER METHODS.

Sample	Specific Surface of Soil, sq. cm. per g	
	Specific Gravity Balance Method	Hydrometer Method
No. 1	8350	7130
No. 2	8085	8260
No. 3	9280	7780
No. 4	5625	4030
No. 5	7670	7830
No. 6	5405	5070
No. 7	7750	7165
No. 8	8715	8275

The average specific surface obtained for the eight samples by the two methods was therefore:

Specific gravity balance method	7610 sq. cm. per g.
Hydrometer method	6943 sq. cm. per g.

It thus appears that the average specific surface obtained by the hydrometer method was 8.76 per cent lower than was obtained by the specific gravity balance method.

Tests were made to determine whether the repeated introduction of the plummet into a suspension changed the test results from what would be obtained if the plummet was only introduced once. A regular test was first run on a sample and the sample was then re-agitated and a reading taken after 9 min.; re-agitated and a reading taken after 18 min., etc. No difference in results was noticed. The possible effect of the vibration of the apparatus due to the motor drive was also investigated and the conclusion was reached that this vibration did not affect the test results.

Specific Surface of Cement:

A 7-g. sample of cement was prepared by combining two U. S. National Bureau of Standards samples with an indicated surface area of 1890 sq. cm. per gram. The result obtained with the chainomatic specific gravity balance was 1883 sq. cm. per gram. Tests have been made on a very limited number of duplicate samples, the specific surface of which had been determined by the Wagner turbidimeter, and the results have been within ±15 sq. cm. per gram.

Observations:

Two motor-driven specific gravity balance apparatuses and an hydraulically operated apparatus have been built.

The first apparatus that was built has a tank to accommodate 20 graduates and a vertical tank speed of 3 cm. per second. It has a lower limit switch and a switch to stop the tank with L = 1 cm. and L = 22 cm. The screw is belt driven and the small switches stop the tank within ±0.2 mm. of the desired elevations. Fluctuations in the cycles and power line voltage cause practically all of the observed error in the elevations. The time schedule for testing 14 soil samples in this apparatus is shown in Table VI.

It will be seen from this schedule that 14 samples are tested in 103 min. or with an average time per sample of 7.36 min.

The second apparatus has a tank to accommodate five samples, a vertical tank speed of 6 cm. per second and small switches to stop the tank with L = 1, 6, 10, 14, 18, and 22 cm. The tank for this apparatus is a one-piece aluminum casting with all surfaces turned in a lathe. The graduate supports are cast aluminum and were machined to the required dimensions. The motor is gear connected to the screw and the gears operate immersed in light oil. Due to the variations in

TABLE VI.—TIME SCHEDULE.

Two minutes before test is to start, begin dispersing sample No. 1 and disperse a total of 7 samples at 1-min. intervals.

2 min. to 8 min. inclusive—first set of readings for samples 1 to 7 (L = 22, T = 2 min.)

9 min. to 15 min. inclusive—second set of readings for samples 1 to 7 (L = 22, T = 9 min.)

18 min. to 24 min. inclusive—third set of readings for samples 1 to 7 (L = 1, T = 18 min.)

23 min.—begin dispersing sample 8 and disperse a total of seven samples at 1-min. intervals.

27 min. to 33 min. inclusive—first set of readings for samples 8 to 14 (L = 22, T = 2 min.)

34 min. to 40 min. inclusive—second set of readings for samples 8 to 14 (L = 22, T = 9 min.)

43 min. to 49 min. inclusive—third set of readings for samples 8 to 14 (L = 1, T = 18 min.)

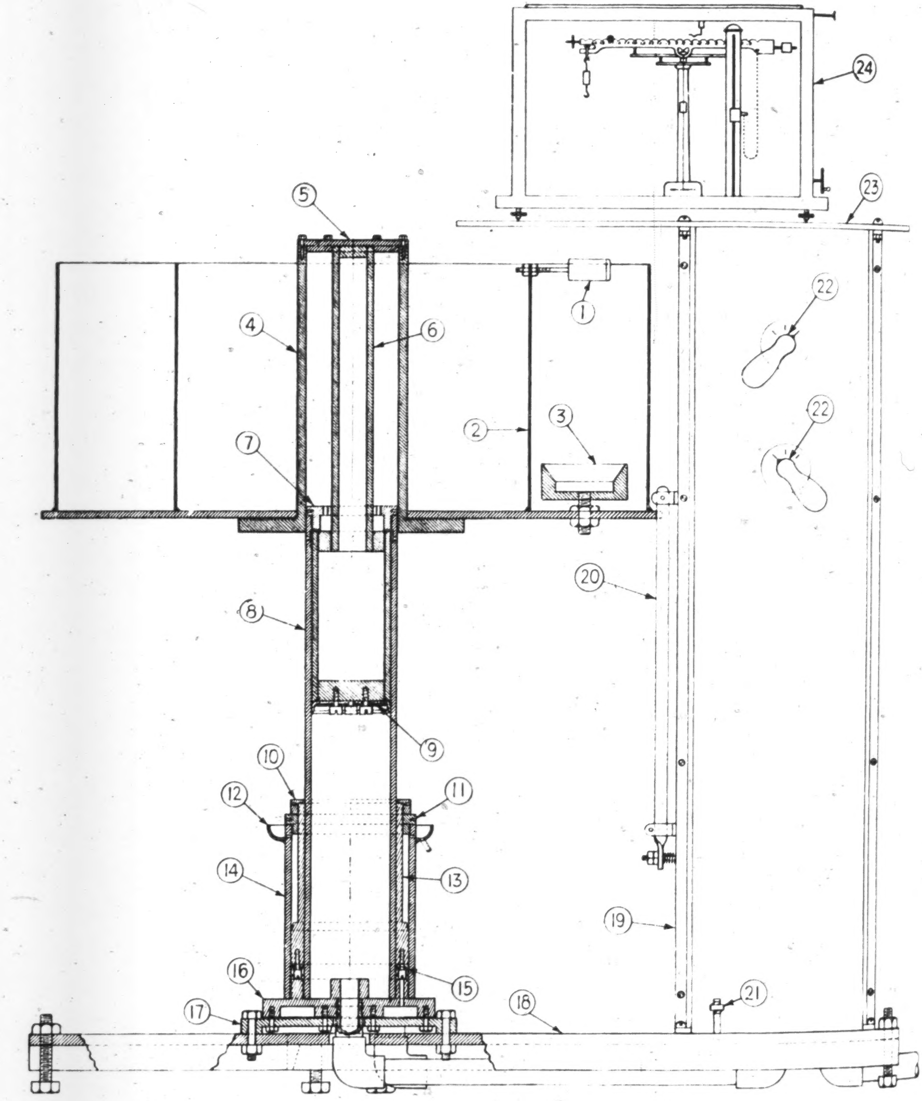

FIG. 9.—Details of Hydraulically Operated Apparatus.

1—Snap clamp for graduates
2—Tank
3—Graduate support
4—Tank sleeve
5—Tank sleeve cap
6—Inner piston
7—Adjustable stop for inner piston
8—Inner cylinder
9—Metal disk to hold leather packing
10—Adjustable cap on outer piston
11—Adjustable stop for outer piston
12—Trough to catch oil or water leakage
13—Outer piston
14—Outer cylinder
15—Metal disk to hold leather packing
16—Upper base for cylinders
17—Lower base for cylinders
18—Base for apparatus
19—Balance support column
20—Vertical guide for slots in lower rim of tank
21—Foot pedal to disengage guide
22—Three-way valves
23—Balance support plate
24—Chainomatic specific gravity balance

64 min. to 70 min. inclusive—fourth set of readings for samples 1 to 7 (L = 22, T = 64 min.)

72 min. to 78 min. inclusive—fifth set of readings for samples 1 to 7 (L = 1, T = 72 min.)

89 min. to 95 min. inclusive—fourth set of readings for samples 8 to 14 (L = 22, T = 64 min.)

97 min. to 103 min. inclusive—fifth set of readings for samples 8 to 14 (L = 1, T = 72 min.)

the cycles and voltage of the electricity furnished to the laboratory it has been impossible to stop the tank of this apparatus within ±0.2 mm. of the required elevations. For this reason no tests are made with this apparatus.

The third apparatus is hydraulically operated and has a tank to accommodate 15 graduates. The tank is raised and lowered by means of two pistons operating in concentric cylinders (see Fig. 9). The upper position of each piston is fixed by means of an adjustable stop and the hydraulic pressure holds the piston in contact with the stop while a reading is being taken. The inner piston raises the tank so that L = 22 cm. and the outer piston so that L = 1 cm. Each cylinder is connected by a tube to a three-way valve mounted in the balance support column. These three-way valves permit raising or lowering either piston at any desired speed. We prefer to operate at a vertical speed of 2 cm. per sec. The apparatus is designed to work with pressures of from 50 to 100 psi. and can operate with either oil or water. The pressure can be supplied by either a pump or direct connection to the city water supply. If water is used, the apparatus should be constructed of brass. The use of this hydraulic apparatus eliminates all vibration and permits stopping the tank at exactly the desired elevations.

If an apparatus were built with a tank to accommodate 29 graduates and with a vertical tank speed of 3 cm. per second it would be possible to disperse samples at $\frac{1}{2}$-min. intervals and test 28 samples in 103 min., thus reducing the average time per sample to 3.68 min.

Precautions:

1. The spherical plummet must be highly polished. If a plummet is used which is not highly polished, results will be erratic due to a film of air on the rough surface.

2. The plummet should be immersed in a graduate containing the liquid medium at least 10 min. before the test is to begin, and it should always be immersed in this graduate when the time interval between readings is more than 1 min. For this purpose the tank should be of such a size that it will hold one more graduate than is required for the test schedule. This graduate also serves for checking the balance reading with the plummet in the liquid medium.

3. The plummet must be washed with the liquid medium after every reading is taken and the plummet should be frequently thoroughly cleaned with CCl_4.

4. The value of L should be checked at intervals. It is convenient to check with L = 1 and checking is done by placing a cork in a graduate with the flat top of the cork horizontal and 1 cm. plus half the vertical diameter of the plummet below the 250-ml. mark on the graduate. The tank is then raised to the required elevation and the length of the hook is adjusted until the balance pointer indicates that the bottom of the plummet has just made contact with the cork.

5. Allowance must be made for the height of the meniscus and the graduates should be filled until the top of the meniscus is the required distance above the 250-ml. mark.

6. The specific gravity balance reading, with the plummet in air and in the liquid medium, should be checked before a test begins.

7. The apparatus should be located in a place where there are no drafts.

8. The balance should always be operated by the same technician. Several days of practice are required before a man becomes reasonably proficient and several weeks before he can achieve maximum efficiency.

9. The tank should always be raised so that not more than 7 sec. will elapse between the time the tank reaches the required elevation and the time the reading is taken. This minimizes the number of particles that can settle on the plummet. An experienced operator can make a reading in 3 sec.

10. If a motor-driven apparatus is used, the water in the tank should always be maintained at the same level. If this is not done the inertia effect of the motor will result in an error in the elevation at which the small switches stop the tank.

11. The temperature of the water in the tank should be maintained at 25 ± 0.2 C. throughout the test. This is done by manually adding warm or cool water. The water in the tank is automatically stirred every time the tank is turned since the graduates are forced through the practically stationary water.

12. The lower limit switch, which automatically stops the tank in the down position, should be so placed that there will be a clearance of at least 3 cm. between the bottom of the plummet and the top of the highest graduate when the tank is down.

Acknowledgment:

The method described in this paper was developed in the Materials Testing Laboratory of the Ministry of Public Works of Venezuela in Caracas. The author is indebted to José Baldó and Leopoldo Turco Rivas for assistance in designing and constructing the apparatus and to Pedro Nicolás Díaz, Eudoro Santiago López, Jaime de Elguezabal, José Baldó, Carlos Luis Pacanins, José Ladislao Andara, and Luis Pantin for assistance in calibrating the apparatus, testing samples, and computing the required tables.

Acknowledgment is made to Francisco J. Sucre, Director of Ways of Communication of the Ministry of Public Works until October, 1945, for permission to develop the method and for encouragement during the extended time of its development. Acknowledgment is also made to Otto Luis Pérez, the present Director of Ways of Communication, for his co-operation in the construction of the hydraulic apparatus and for permission to present this paper.

DISCUSSION

MR. ALAN R. LUKENS.[1]—We have used a method similar to the one described by Mr. Barrett, also employing the chainomatic balance to make sedimentation tests. Thus we are in a position to appreciate the clever work done by Mr. Barrett. His ingenuity has overcome many mechanical difficulties and he has made it easy to conduct multiple tests.

We note Mr. Barrett calculates his specific surface from the particle size distribution and then uses the Wagner surface areas as a means of correlation. We have made numerous comparisons of data obtained from the Wagner turbidimeter with surface area data we obtain with our Brunauer-Emmett-Teller nitrogen-adsorption apparatus. We find in many cases the latter shows decidedly higher figures, particularly when the particle has the somewhat porous structure common to portland cements. Thus we recommend the shape and the structure of particles comprising a distribution be considered when one seeks accuracy in calculating specific surfaces from the distribution of sizes.

Mr. Barrett would be wise to take into account entrapped air introduced by brush stirring. This air as it rises alters the velocity of sedimentation and changes the specific gravity at points below the surface, particularly at the distance at which he makes certain of his tests.

Many of these causes of inaccuracies we have overcome but only as the result of about fifteen years of constant work. We use in conjunction with the chainomatic balance a specially designed centrifuge and by this means obtain particle size distributions down to somewhat less than 0.1 μ, in a matter of hours.

When testing soils, we find each must receive careful preliminary examination so as to select that dispersing agent particularly suited to it. Sedimentation tests are of value only after perfect dispersion has been achieved.

Mr. Barrett has done a splendid job in developing his method of uniformly and carefully introducing his plummet; his tables surely will be useful in routine laboratories. We congratulate him upon his ingenuity and upon the method of presenting his paper to us.

MR. LINCOLN T. WORK.[2]—The author is to be commended on the development of an apparatus which points in the direction of speed. I should, however, indicate that the hydrometer method, if scheduled in a work day, can be carried out in less time than one would normally think. In other words, if the test program begins around 2 or 3 o'clock in the afternoon and the last reading is taken in the morning, the time during the night, which is not useful in productive effort, is used to advantage.

I have, however, two or three questions which might be answered now or in subsequent work. One is the question of correlation of sieve diameters with the settling diameters. There is considerable information on the subject, but

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[1] Vice-President, Powdered Material Research Laboratories, Cambridge, Mass.

[2] Director of Research and Development, Metal and Thermit Corp., Woodbridge, N. J.

I did notice that the sieve separation and the settling tests were carried out on the same soils and did not notice any correlation of the two types of diameters.

With respect to the absorption method, to which Mr. Lukens referred, I am wondering whether we know the properties of cements or soils with respect to what might be considered internal surface which would not be recorded on the sedimentation but would be recorded on the absorption method. Irregularities of external surface, however, do probably represent an error from the conventional which we assume. Also I raise a question in connection with the paper about the effective use of 1-cm. depth. It is a great help in getting speed, but the size of the ball, having a radius of 0.78 cm. brings the top of the ball rather close to the surface and the bottom of the ball some distance down so the 1-cm. center of gravity point does not represent the effective depth.

In other words, the diameter of the ball in relation to the depth at which it is used, in that case, may present a dangerous situation. I presume it does not make much difference on soils. Otherwise, I think the problems are those which will develop as we see more and more work being done on this type of apparatus.

A MEMBER.—Were any comparisons made?

MR. E. V. BARRETT (*author's closure*).—Mr. Lukens states that the surface area obtained with the Brunauer-Emmett-Teller nitrogen-absortion apparatus is in many cases decidedly higher than the surface area obtained with the Wagner turbidimeter. The nitrogen-absortion apparatus measures both exterior and internal surface and, since all cement has a somewhat porous structure, the surface area of the voids in the particles would normally amount to an appreciable area; therefore, any result obtained would be in error by the amount of this internal area. Furthermore, there appears to be no question that the surface areas obtained with the Wagner turbidimeter are lower than they should be. This is due to the fact that the smallest particle for which a turbidity reading is taken is 7.5 μ. When it is considered that if a gram of cement has 1 per cent of particles with an average size of 0.0075 mm., the surface area is 25.4 sq. cm. while 1 per cent of particles with an average size of 0.001 mm. has a surface area of 190.5 sq. cm., it can readily be seen that the specific surface determined by the Wagner turbidimeter can be considerably lower than the true surface.

Since the A.S.T.M. had adopted the turbidimeter in the Standard Method of Test for Fineness of Portland Cement by the Turbidimeter (C 115 – 42),[3] a procedure was developed whereby a comparable result could be obtained using the chainomatic specific gravity balance. In developing this procedure, the specific surfaces of a number of samples of cement were determined with the Wagner turbidimeter. Samples of the same cements were then tested with the specific gravity balance and the surface area of all particles 0.005 mm. or larger was computed for each sample. On the basis of the difference between the surface areas obtained in this way and the surface areas obtained with the Wagner turbidimeter it was then possible to compute the average size of particles smaller than 0.005 mm. that would result in a specific surface equal to that obtained with the turbidimeter. It was found that the required particle size was 0.0035 mm. The author expects in the near future to make a further investigation of the specific surface of portland cement with the specific gravity balance method, determining particle size to 1 μ and assuming the average size of particles smaller than 1 μ on the basis of the best information available.

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[3] 1944 Book of A.S.T.M. Standards, Part II, p. 47.

Mr. Lukens' remarks relative to terminal velocities and entrapped air would, I believe, apply primarily to research and would not apply to routine testing. The largest soil particle for which a reading is taken at 1 cm. below the surface is 3 μ and the reading is at 18 min. Gessner[4] has demonstrated that the time required for a particle to reach its terminal velocity is only appreciable for extremely coarse particles, a conservative lower limit for which would be 100 μ. Relative to the entrapped air, the first reading is purposely taken at 2 min. and the specific gravity of the suspension is determined at a depth of 22 cm. since we have found that under these conditions our results are not affected by the rising air bubbles. It has been our experience that the principal error caused by the small rising air bubbles is due to the decreased density of the solution caused by the bubbles and to a lesser extent the impact of the rising air bubbles against the plummet. It is believed that entrapped air could be reduced to a minimum by using an air-free liquid medium and dispersing in a vacuum.

The chainomatic specific gravity balance method was developed for the rapid routine testing of soil samples and practically all of our work with this method is in testing soil samples from dams, airports, highways, etc. We have found that practically all flocculation of dispersed soil samples is due to the presence of salts and other soluble material and that the washing of a soil sample, in accordance with our procedure, reduces the salts and soluble material to a point where flocculation does not occur.

Mr. Work seems to have the impression that the only advantage of the method covered in my paper is speed, and it is unfortunate that he has this impression. We have made several thousand hydrometer tests and it was complete dissatisfaction with the hydrometer method that led to the development of the chainomatic specific gravity balance method. The hydrometer method requires 24 hr. for a determination of the percentage of soil particles finer than 1.5 μ and in addition grossly violates one of the fundamental assumptions on which all sedimentation tests are based since the hydrometer, instead of indicating the density of a suspension at some plane below the surface, indicates the average density of a column of dispersion at least 15 cm. high. It can only be by pure accident that the density indicated by the hydrometer will correspond to the actual density at the assumed plane.

We found, for soil samples, that when working with the hydrometer and plotting particle size on the basis of the combined sample, a projection of that portion of the curve representing percentage finer as determined by sieves failed to meet the curve as determined by the hydrometer, by from 1 to 7 per cent. However, in the case of all of the hundreds of soil samples that we have tested with the specific gravity balance method the two curves meet exactly.

Mr. Work has raised a question about the density determinations made at 1 cm. below the original surface of a suspension. The radius of the spherical plummet is about 0.78 cm. and the volume of the plummet is 2 cu. cm. The introduction of the plummet into a suspension raises the level of the suspension by 2 mm. Since the density of the suspension is determined at a depth of 1 cm. below the original surface of the suspension, the center of the plummet is then 1.2 cm. below the existing surface at the time the density is determined. It therefore follows that the top of the plummet is 0.42 cm. below the surface when the density is determined. Under these conditions there can be no difference between determining the density of a suspension at a depth of 1 cm. below the original surface or 22 cm. below the original surface and the center of the plummet will in both cases represent the effective depth at which the density is determined for soils or for any other finely divided particles.

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[4] H. Gessner, "Die Schlammanalyse," Akademische Verlagsgesellschaft, Leipzig (1931), pp. 12–14.

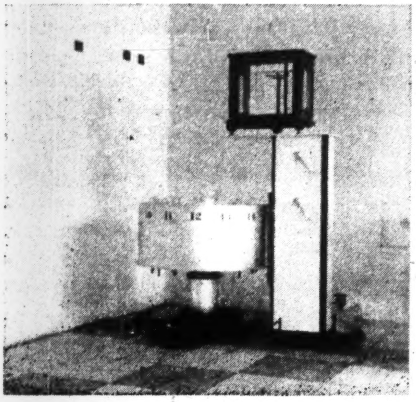

FIG. 10.—Hydraulically Operated Apparatus with Tank in Down Position.

In answer to the question about comparisons, comparative tests have not been made with the chainomatic specific gravity balance method and either the Lea and Nurse or the Blaine air permeability apparatus. In the author's opinion the inherent errors of these methods due to the adhering air film surrounding each particle, the variable permeability of the filter paper, and the practical impossibility of exactly controlling the porosity of the bed of cement must result in an appreciable error. In addition, there does not seem to be agreement as to the method that should be used in making the computations.

We have discontinued use of the motor-driven apparatus and make all of our tests with the hydraulically operated apparatus shown in Fig. 9 of the paper and the accompanying Fig. 10. The apparatus operates with No. 20 oil and pressure is supplied from a small pump discharging into a 15-gal. hot water tank. A pressure regulator mounted on the tank automatically cuts the pump motor in at 50 psi. and cuts it out at 75 psi. Gate valves mounted in the pressure and discharge lines to the apparatus permit raising and lowering the tank at any desired vertical velocity. The discharge line has a connection to each of the two cylinders and a check valve is mounted in each of these connecting lines. These check valves prevent back pressure from raising one piston while oil is being released from the other cylinder. The discharge line discharges into a 15-gal. hot water tank that is used as a sump and the pump draws the oil from this sump.

LABORATORY INVESTIGATION OF ANTI-STRIPPING ADMIXTURES USED FOR PROMOTING WETTING POWER AND ADHESION BETWEEN BITUMENS AND AGGREGATES*

By John C. Sprague[1]

Synopsis

The general relationship between, and effect of anti-stripping admixtures upon, the wetting power (as opposed to coverage), adhesion (or stripping resistance), and stability of bituminous mixtures in the presence of water have been investigated and the results reported and discussed in this paper. It is pointed out that coverage of an aggregate with bitumen may often be obtained without wetting, but that thorough wetting of an aggregate is essential to good adhesion. The results suggest that "hydrophilic" and "hydrophobic" are relative rather than absolute terms, as used in bituminous construction, and that they should not be applied unreservedly to an aggregate for all conditions of use. Findings based upon a conventional static immersion stripping test, and upon a stability test of soaked compared with unsoaked specimens are presented, and discussed.

As a result of the laboratory phase of the investigation, it is found that none of the nine anti-stripping admixtures tested can be said to have an over-all efficiency, for the range of nine aggregates (from a dense granite to a porous coral) and three bitumens included in the program, from the standpoint of promoting wetting power, stripping resistance, and stability retention of plant-type bituminous mixtures. None of the admixtures would promote wetting power of asphalt (AC-8) when mixed with surface-wet graded aggregates; nor would any of them sufficiently promote the wetting power of any of the bitumens tested when mixed with the surface-wet, porous lime rocks.

On the other hand, practically all of the admixtures were quite effective with the surface-dry aggregates; and several of the admixtures were very effective with several of the surface-wet aggregates in promoting wetting power, stripping resistance, and stability.

Upon request of the Engineer Board, Fort Belvoir, Va., and approval by the Chief of Engineers, Corps of Engineers, U. S. Army, the Division Engineer, South Atlantic Division, directed the Division Materials Testing Laboratory to conduct appropriate tests to determine the efficacy of certain anti-stripping admixtures used in bituminous mixtures for promoting wetting power and anti-stripping properties of bitumens when used with wet, hydrophilic aggregates. The purpose of the program was to try to find an additive which would be effective with the normal range of bitumens in use in certain theaters of war, to the end that they would coat, and adhere to, a wide variety of wet aggregates. The laboratory phase of the test program was initiated in April, 1945, and was to be followed by field pilot tests. However, the laboratory tests were not completed before cessation of hostilities and, in light of removal of the emergency, the "follow-up" field tests have been postponed indefinitely.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] Engineer in Charge, Division Materials Testing Lab., South Atlantic Division, Corps of Engineers, U. S. Army, Atlanta, Ga.

This report covers the results of a laboratory investigation on the effect of each of nine additives upon the wetting power, stripping characteristics, and stability retention of three asphalts (AC-8, RC-2, and MC-2) combined in bituminous mixtures with nine different aggregates.

EXPLANATION OF TERMS

Adhesion: Used interchangeably with the expression "Anti-Stripping"; power of attraction between liquid bitumen and aggregate.

Blank: Plain bitumen, without admixture.

Blend: Plain bitumen blended with admixture.

Consistency: Of bitumen, as measured by viscosity, penetration and/or softening point.

Coverage: Merely covering or coating an aggregate particle with a film of bitumen, without it necessarily adhering firmly to the particles.

Hydrophilic: Greater attraction of aggregate for water than for oil (water loving).

Hydrophobic: Greater attraction of aggregate for oil than for water (water hating).

Wettability: Susceptibility of an aggregate to being wetted by a bitumen.

Wetting Power: Ability of a bitumen to cover (or coat), and adhere firmly to an aggregate (as opposed to coverage).

MATERIALS

The following materials were involved in this investigation:

Aggregates:

Crushed gravel (predominately flint); Feldspathic quartzite; pit-run gravel (predominately quartz); granite (granodiorite, having definite granitoid texture); Brooksville lime rock (predominately $CaCO_3$ and $MgCO_3$); reef coral; argillaceous limestone; Ojus lime rock (predominately $CaCO_3$ and $MgCO_3$, with appreciable SiO_2); quartz sand, coated with iron oxide.

Bitumens:

Asphalt cement, 85 to 100 penetration (AC-8);[2] rapid curing cut-back asphalt, 100 to 200 viscosity range (RC-2)[3] and medium curing cut-back asphalt, 100 to 200 viscosity range (MC-2).[4]

Anti-Stripping Admixtures:

Nine additives, designated A, B, C, D, E, F, G, H, and I, furnished by leading processors in the country, were investigated to determine their efficacy as wetting and anti-stripping agents without, at the same time, having an adverse effect upon other characteristics of bituminous mixtures. A brief description of each of the additives follows:

Agent A.—An oleic amine, greenish in color, of consistency such that it will not flow when cool, but will become a liquid under warm-weather conditions.

Agent B.—A mixture of organic bases and other organic compounds. It is light tan in color and at normal temperature has the consistency of cup grease.

Agent C.—An alkylated oleic amine, dark brown, having the consistency at normal temperature of very heavy motor oil.

Agent D.—Essentially a fatty acid in combination with an amine, to which has been added aluminum sulfate and a tall oil type soap, all mixed to a given dilution with a cut-back asphalt. It is brown in color, having a consistency at normal temperature of molasses.

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[2] Standard Specifications for Asphalt Cement, 1942 Book of Am. Assn. State Highway Officials, Part I, p. 5.

[3] Tentative Specifications for Cut-Back Asphalt (Rapid Curing Type) (D 597 – 40 T), 1944 Book of A.S.T.M. Standards, Part II, p. 1372.

[4] Tentative Specifications for Cut-Back Asphalt (Medium Curing Type) (D 598 – 40 T), 1944 Book of A.S.T.M. Standards, Part II, p. 1374.

Agent E.—Essentially the stearyl amine salt of the stearyl amide of ethyl phosphoric acid. It is a fat-like tan solid at room temperature and is melted to an amber oil before blending with a bitumen.

Agent F.—Essentially the same as that of agent D, except that it is diluted with cut-back asphalt in different proportions, and except that it is milled with water which serves as a binder. It is dark brown and has the consistency at normal temperature of light grease.

Agent G.—A soap obtainable from petroleum sources, not readily subject to being incorporated directly into a bitumen in its pure or 100 per cent concentrated form. For this investigation it was furnished as a 10 per cent concentrate in cut-back asphalt (RC-2), compounded to the viscosity range of regular RC-2 cut-back asphalt. Therefore, when it is used with other types of bitumen, it naturally tends to change their physical characteristics. It decreases the consistency of the AC-8 asphalt.

Agent H.—A fatty oleic poly-amine, light tan in color and having the consistency at room temperature of heavy axle grease. The additive has to be heated in order to mix it with bituminous materials.

Agent I.—A heavy metal soap, light tan in color. Its consistency at normal temperature is that of cup grease.

PROCEDURES

Modified Oberbach Stripping Test:[5]

The modified Oberbach Stripping Test (static immersion test) was used to measure the stripping, as such, of bituminous films from the surfaces of the aggregates. It was performed, with companion samples, under the following stipulated conditions:

1. Immediately after mixing, a sample was immersed in distilled water at 77 F. After a 24-hr. immersion period, the sample was examined visually to determine the extent of stripping that had occurred.

2. A sample was cured at a temperature of 77 F. for 24 hr., then immersed in distilled water at 100 F., and after a period of 24-hr. immersion, examined as stated above.

3. A sample was cured in an oven at 140 F. for 24 hr., then immersed in distilled water at 140 F. for 24 hr. and examined as stated above.

4. Selected mixes were subjected to two cycles of freezing and thawing, after which they were subjected to stripping action as described in (1) and (2) above. The AC-8 asphalt mixes received no preliminary curing, but the cut-back asphalt mixes were cured for 24 hr. at 77 F. prior to testing. In a few instances, companion samples were subjected to 25 cycles of freezing and thawing with no different results than were obtained with two cycles.

This static immersion stripping test is similar to several others which have been used. After the mixes had been prepared, as described, containers having a capacity of 200 ml. were filled one third to one half with mixed material and then the rest of the way with distilled water. After the 24-hr. period at 77 F. the mixes were visually inspected. In order to minimize the personal equation, and variations such as color of aggregate, optical effects due to variable light intensities, and other natural phenomena, estimations of area stripped were made on submerged samples under constant light conditions, by two operators making independent estimates. In this phase of the investigation, stripping tests were performed in groups, each group

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[5] Conforms with procedure outlined in unpublished report of the Engineer Board, Fort Belvoir, Va.

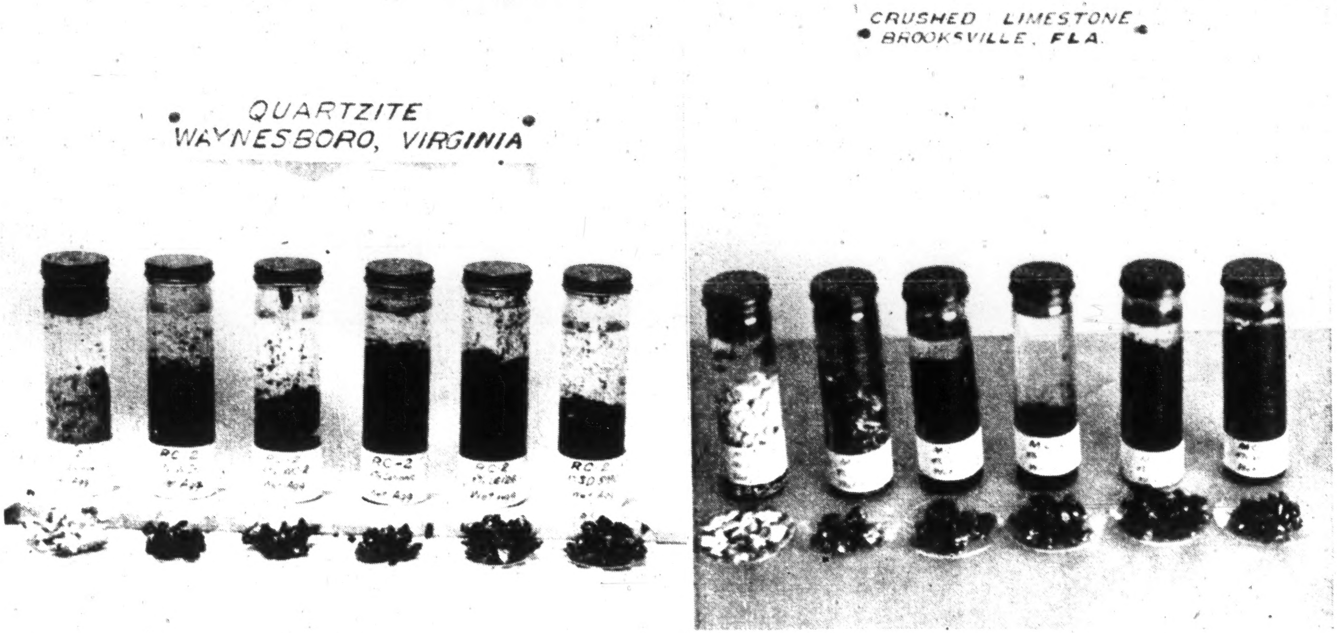
QUARTZITE
WAYNESBORO, VIRGINIA
CRUSHED LIMESTONE
BROOKSVILLE, FLA.

FIG. 1.—Typical Views of Surface-Wet Siliceous and Calcareous Aggregate Mixes After Completion of the Modified Oberbach Stripping Test.
Sample at left shows mix with no admixture (blank). The other samples show mixes with admixtures taken at random.

consisting of one aggregate and one moisture condition of aggregate, the blank sample (bitumen without admixture) in each instance being directly comparable with the blends (bitumen with admixture) of that group. Data recorded in the performance of this visual inspection included condition of coating after mixing, estimate of percentage of area uncoated, estimate of areas only thinly coated, and type of stripping (whether globular, partial release of bituminous material, or complete release of bitumen from the surface of the aggregate). It was considered that stripping was not excessive if it amounted to less than 25 per cent. Figures 1 and 2 show the types of containers used during the immersion and curing stages of the test.

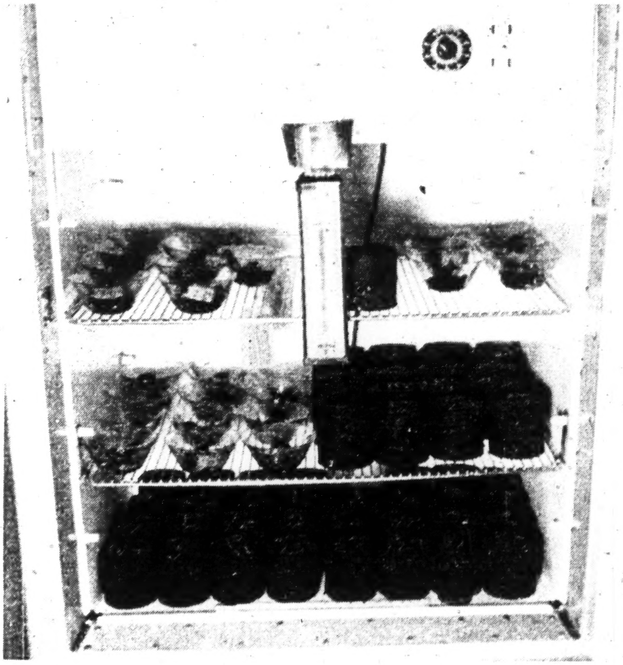

FIG. 2.—Forced Draft Constant Temperature Cabinet Used for Curing Oberbach Stripping Test Specimens; and for Bringing Stability Specimens to Constant Temperature of 77 F. Before Placing in Compression Machine.

The size aggregate used in this phase of the investigation ranged from $\frac{3}{4}$ in. to No. 4, with all material above and below these limits screened out. Aggregates which were tested in a surface-wet state were soaked for 24 hr. prior to mixing. Before being placed in the mixing bowl, all wet aggregates were shaken under a constant condition in a wire mesh basket until substantially all free-flowing water had been drained off, after which the percentage of surface moisture was determined (the surface moisture ranged from a low of 1.6 for pit-run gravel to a high of 2.8 for Ojus lime rock, and 12 for sand). Aggregates used in a surface-dry condition were at room temperature when mixed, except that aggregate mixed with the AC-8 asphalt had to be heated to about 150 F.

Initially, a small Hobart, kitchen-type mixer fitted with a special paddle was tried for the mixing operation. This was soon dispensed with, however, in favor of mixing by hand, as it was found that the latter method was faster and better suited to coat the aggregate uniformly with a film of bitumen. The percentage of bitumen used was such that the film thickness on the aggregate particles was substantially constant for all mixtures, the optimum in each case depending upon the physical characteristics of the aggregates.

Axial-Compression Stability Test:

Suggestions made by the Public Roads Administration[6] furnished the stimulus for developing and applying this type of test to the problem of evaluating additives used to promote the wetting power and anti-stripping properties of bitumens. At the time the method was initiated, it was considered particularly useful for evaluating those mixtures where stripping induced by the conventional methods was not a visual physical actuality, yet where there might be incipient stripping, that is, where the film of bitumen might be covering the aggregate but not adhering tightly to it. It is essentially a stability test in which the stability of a bituminous mixture is measured by unconfined compression along the axis of a cylindrical specimen. In order to simulate field conditions, companion specimens are tested for stability in an unsoaked state, and after having been soaked for prescribed periods of time. See the appendix for further discussion of this method of test for evaluating stripping tendencies between bitumens and aggregates, and for details covering the development of the following procedure which was adopted:

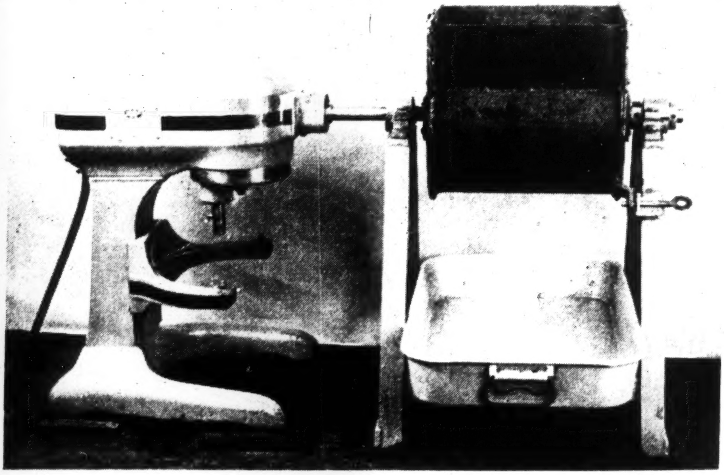

FIG. 3.—View of Laboratory Pugmill Used in Preparing Bituminous Mixes.

1. Aggregates were synthetically graded from $\frac{3}{4}$ in. to No. 80 (Table I), and 4000-g. batches weighed out. Surface-wet aggregates were soaked for 24 hr., drained of excess free water, and per cent surface moisture determined before they were placed in the mix. (Surface moisture ranged from 9 to 12 per cent, depending upon character of the aggregate.)

2. All batches were mixed in a small

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[6] From unpublished data, subsequently published in *Public Roads*, Vol. 24, No. 5, under title "A Test for Determining the Effect of Water on Bituminous Mixtures," by J. T. Pauls and H. M. Rex.

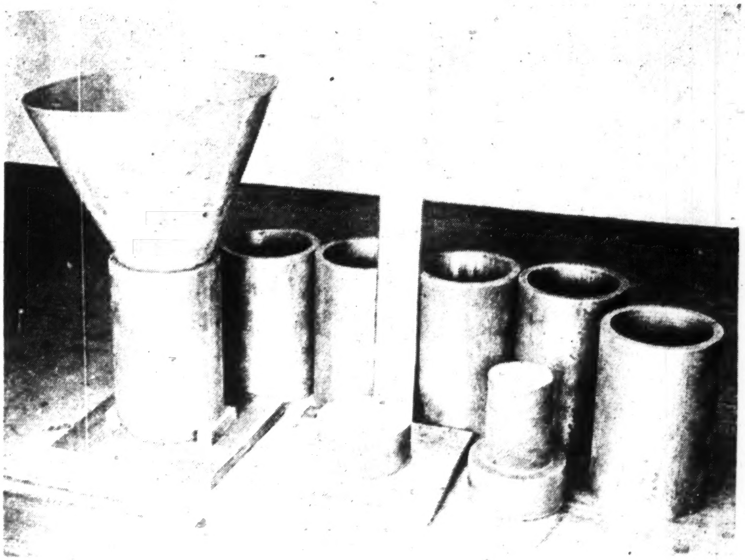

FIG. 4.—View of 3-in. Specimen Molds.

laboratory single pugmill (Fig. 3) for approximately two minutes or until the aggregate was coated, if it could be coated. The bitumens were heated in accordance with standard practice, as was the dry aggregate which was mixed with AC-8 asphalt. AC-8 asphalt mixtures were molded immediately after mixing and placed in a forced draft cabinet at 77 F. RC-2 and MC-2 cutback asphalt mixes were cured at a temperature of 140 F. in a forced draft oven, respectively for 24 and 48 hr., after which they were brought to room temperature, molded, and placed in the constant temperature cabinet at 77 F. Figure 2 shows a view of molded specimens in the cabinet.

TABLE I.—GRADATION OF AGGREGATE FOR BITUMINOUS MIXES.

Sieve Size	Passing, per cent
¾ in.	100
⅜ in.	86
No. 4	62
No. 10	46
No. 40	23
No. 80	0

3. The specimens were compacted to a height of 3 in. in 3-in. cylindrical molds by double plunger action (to insure uniform density), under a load of 2000 psi. for 2 min. Figures 4 and 5 show views of the molds used and of the compacting press.

4. Density and percentage absorption of the specimens were determined by established procedure.

5. Specimens were made in companion sets of four each, half of which were tested for stability in an unsoaked condition and half in a soaked condition. Figure 6 shows the apparatus used for determining the axial-compression stability of the specimens. Specimens to be soaked were first subjected to a 30-cm. vacuum in distilled water for 15 min. and then placed in a water bath for 24 or 48 hr. Figure 7 shows the vacuum apparatus used.

6. Test results were evaluated in terms of stability losses or gains between blank as compared with blended mixes, and between soaked *versus* unsoaked

specimens. See appendix for detailed explanation.

DISCUSSION

Since the physical characteristics of the materials involved in this investigation tend to have a significant effect upon the finished mixes, the more pertinent characteristics were determined as recorded in Tables II to V.

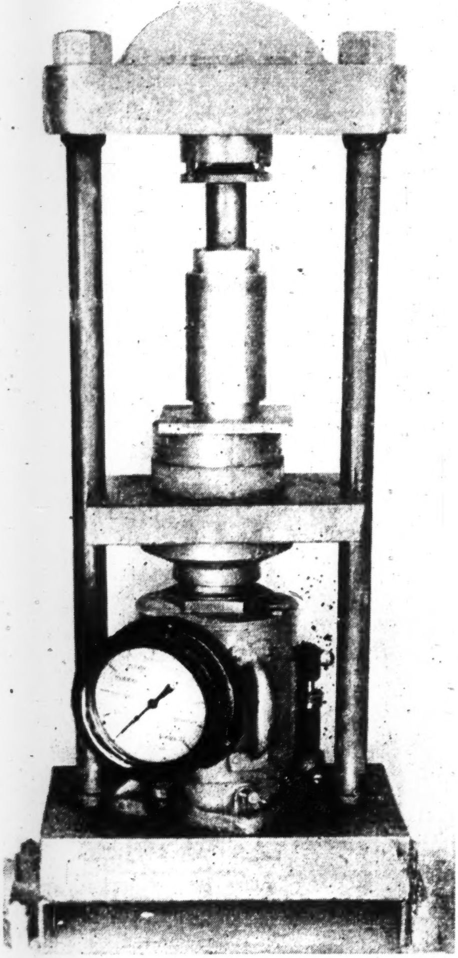

FIG. 5.—Press for Compacting 3-in. Specimens Simultaneously at Both Ends, Using Double Plunger Method.

It is considered that absorption, surface texture, and predominating minerals are the three characteristics which have major bearing on capacity of the aggregates for wettability and for holding a bituminous film in the presence of moisture. For example, it will be observed elsewhere in the report that those aggregates having low absorption are more susceptible to wetting than are the more porous aggregates, and that the wet, porous lime rocks could not be coated at all with the bitumens used. Surface texture is also a factor bearing upon wettability of the solid, a smooth surface being more readily wetted than a grainy one. On the other hand, the siliceous aggregates showed a greater affinity for water than for asphalt as evidenced by stripping tendencies, whereas the limestone and lime rocks tended to be at least feebly hydrophobic and to have greater affinity for asphalt, regardless of surface texture. This follows the tenet that, in general, acid minerals tend to be hydrophilic and base minerals, hydrophobic. Although a smooth surface may be more readily wetted, a rough texture surface and/or a porous surface is apt to produce greater adhesion once the surface is wetted by the bitumen. It is evident that some of the factors which promote wetting are not advantageous from standpoint of adhesion, and a number of factors which tend to retard wetting may greatly increase adhesion. See Table II for physical characteristics of the aggregates.

The effect of admixtures on the bitumens is shown in the physical analysis of bitumens and bituminous blends in Tables III, IV, and V. It will be noted that penetration and viscosity were the important characteristics most affected by the additives, the penetration of blends being increased in the majority of cases to an extent which failed specified limits; and viscosity in most cases

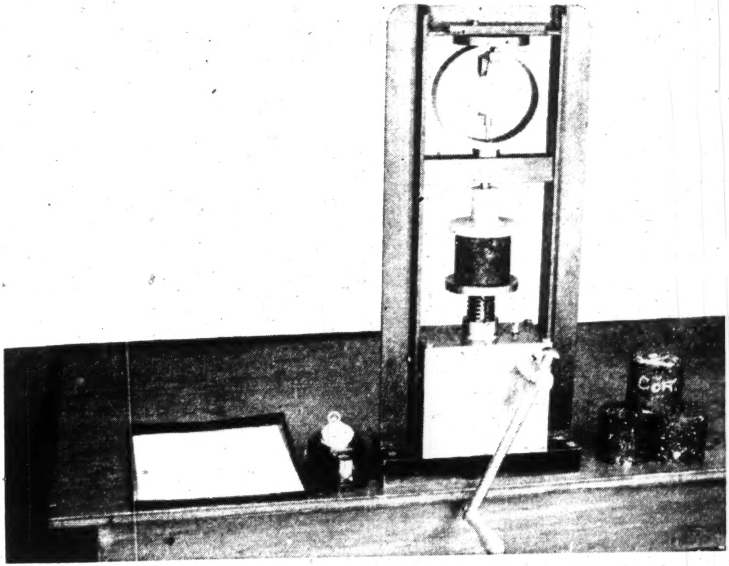

FIG. 6.—Compression Machine for Measuring Stability of 3-in. Specimens.

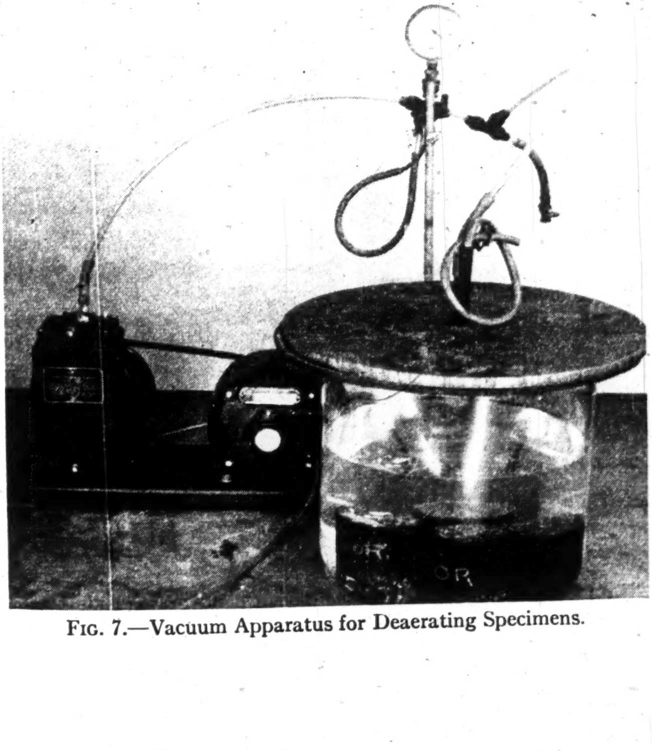

FIG. 7.—Vacuum Apparatus for Deaerating Specimens.

TABLE II.—PHYSICAL CHARACTERISTICS OF AGGREGATES

Aggregate	Specific Gravity						Absorption, per cent		Los Angeles Abrasion, Loss, per cent	Predominately	Surface Texture
	Bulk (dry)		Bulk (saturated)		Apparent						
	Coarse	Fine	Coarse	Fine	Coarse	Fine	Coarse	Fine			
Limestone	2.69	2.57	2.70	2.60	2.73	2.67	0.5	1.4	19.6	Calcareous	Smooth and dense
Granite	2.66	2.52	2.67	2.66	2.69	2.72	0.5	0.7	34.9	Siliceous	Granitoid crystals are smooth
Pit-run gravel	2.59	2.62	2.61	2.65	2.64	2.69	0.7	0.8	50.4	Siliceous	Fairly rounded and smooth
Quartzite	2.54	2.50	2.57	2.54	2.61	2.60	1.1	1.5	27.4	Siliceous	Grainy
Crushed gravel	2.38	2.42	2.46	2.50	2.57	2.57	2.9	2.0	24.1	Siliceous	Smooth and porous
Lime rock (Brooksville)	2.44	2.51	2.51	2.55	2.63	2.62	3.0	2.2	34.3	Calcareous	Smooth-coated porous
Lime rock (Ojus)	2.14	2.44	2.28	2.48	2.44	2.55	5.7	2.3	36.9	Calcareous	Rough—sponge-like
Reef coral	2.06	2.30	2.19	2.45	2.36	2.55	6.2	4.5	37.0	Calcareous	Rough—sponge-like
Sand	...	2.55	...	2.59	...	2.65	...	1.4	...	Siliceous	Angular—coated with iron oxide

TABLE III.—ANALYSIS OF AC-8 ASPHALT BLENDED WITH THE VARIOUS ADMIXTURES.

Bitumen	Usual Specification		AC-8	AC-8	AC-8	AC-8	AC-8	AC-8
Additive			Blank	D	D	E	I	G
Additive, per cent	Minimum	Maximum	None	4	33	4	3	3
Specific gravity at 77/77 F.	1.01	...	1.03	1.02	0.99[a]	1.02	1.03	
Flash point by Cleveland Open Cup, deg. Fahr.	400	...	530	520	470	600	510	250[a]
Loss on heating of 50 g. after 5 hr. at 325 F., per cent	...	1	0.06	0.4	0.9	0.1	0.1	5.76[a]
Penetration of 100 g. for 5 sec. at 77 F., cm.	85	100	95	135[a]	305[a]	130[a]	88	305[a]
TESTS OF RESIDUE								
Ductility at 77 F., cm.	100	...	109+	109+	too soft[a]	109+	109+	100
Penetration of 100 g. for 5 sec. at 77 F., per cent	60	...	94.7	99.3	100	100	97.7	46.0[a]
Solubility in CCl_4, per cent	99.5	...	99.8	99.7	99.9	99.8	99.8	99.9
Softening point, Ring and Ball method, deg. Fahr.	100	...	118	108	85[a]	110	114	90
Oliensis spot test	Negative		Neg.	Neg.	Neg.	Neg.	Neg.	Neg.

[a] Fails specified limits, as indicated.

increased, as compared with the blank sample results. This is significant, and reflects itself in a tendency for lowered stabilities of unsoaked specimens made from bituminous mixtures in which blends having higher penetration are used. On the other hand, a few of the blends showed up satisfactorily in the analysis, but showed poor to mediocre performance in the stripping and stability tests. Bitumens of relatively high penetration, which at the same time had high viscosity and which had wetting power, performed quite satisfactorily when worked into mixes which were tested in a soaked condition. High viscosity of a bitumen tends to facilitate adhesion, once wetting has been secured. An asphalt film of low viscosity is much more subject to stripping from an aggregate surface, particularly if the surface is smooth, than is a film of high viscosity.

TABLE IV.—ANALYSIS OF RC-2 CUT-BACK, AND RC-2 BLENDED WITH THE VARIOUS ADMIXTURES.

Bitumen	Usual Specification		RC-2	RC-2	RC-2	RC-2	RC-2	RC-2	RC-2	RC-2	RC-2	RC-2	RC-2	RC-2	RC-2	RC-2	RC-2	RC-2
Name of Additive	Minimum	Maximum	Blank	D	D	E	E	B	B	F	F	G	A			C		H
Additive, per cent			None	1.75	4.0	2.5	4.0	1.25	3.0	2.5	4.0	3.0	1.25	3.0	4.5	1.5	3.0	0.5
Specific gravity at 77/77 F.	...	...	0.951		0.951		0.951		0.949		0.950			0.951				
Flash point by Tag Open Cup, deg. Fahr.	80	...	94	85	97	120	106	106	95	99	102	115	98	90	90	102	109	104
Saybolt furol viscosity at 140 F.	100	200	153	178	165	200	185	188	166	170	150	115	185	180	178	190	200	200
Distillation, total per cent by volume of total distillate																		
to 374 F.	...	...	48.5	45.8	41.1	47.8	47.3	34.7	41.6	50.0	46.9	34.0	37.5	46.8	45.7	44.9	41.0	42.9
to 437 F.	40	...	71.5	68.8	55.7	65.2	66.0	61.2	66.1	69.0	68.9	64.2	66.7	68.1	67.4	65.3	63.0	65.3
to 500 F.	65	...	81.5	81.3	79.6	78.3	82.5	75.5	80.8	81.0	81.7	75.5	79.2	83.0	82.6	79.6	78.0	81.6
to 600 F.	87	...	93.0	93.8	91.9	91.3	91.2	87.8	94.7	92.0	92.7	92.5	91.7	93.6	93.5	93.9	94.0	93.9
Residue from distillation to 680 F. by volume, per cent	67	...	75.5	76.0	75.0	77.0	77.2	75.5	76.5	76.0	75.5	73.5	76.0	76.5	77.0	75.5	75.5	75.5
TESTS OF RESIDUE																		
Ductility at 77 F., cm.	100	...	109+	100+	100+	100+	109+	100+	109+	100+	109+	109+	100+	100+	...	100+	100+	100+
Penetration of 100 g. after 5 sec. at 77 F.	80	120	98	126[a]	156[a]	188[a]	230[a]	118	197[a]	130[a]	137[a]	91	100	143[a]	179[a]	99	106	96
Solubility in CCl_4, per cent	99.5	...	99.9	99.5+	99.9	99.5+	99.9	99.5+	99.8	99.5+	99.9	99.9	99.5+	99.5+	99.5+	99.5+	99.5+	99.5+
Oliensis spot test	Negative		Neg.	Neg.	Neg.	Neg.	Neg.	Neg.	Neg.	Neg.	Neg.	Neg.	Neg.	Neg.	Neg.	Neg.	Neg.	Neg.

[a] Fails specifications, as indicated.

A comparison between the viscosities of the blends and plain bitumens, as applied to the stripping tendency and stability of the bituminous mixtures, brings out this point in several instances.

The additives were mixed with the bitumens, in the percentages indicated before the bituminous blends were incorporated with aggregate in the mixes.

At the time this investigation was initiated, instructions were to expedite it in every manner possible to the end that the results, if favorable, could be used in connection with bituminous pavement construction under adverse conditions in the various theaters of war. In order to obtain the necessary information as quickly as possible, disregarding economic considerations, it was decided to use the maximum percentages of the various additives recommended by their respective manufacturers. The net result was that although practically all of them gave highly satisfactory results as measured by the static stripping test, they were largely a failure from the standpoint of stability of the bituminous mixtures. Subsequently, the situation became less exigent due to cessation of hostilities and more time could be given to developing the test along more realistic lines. Consequently, a sufficient number of preliminary mixes was then prepared for each admixture to determine the minimum amount required with RC-2 cut-back asphalt to obtain wettability of surface-wet aggregates and substantially to arrest stripping action of bituminous films from surface-dry aggregates. RC-2 cut-back asphalt was selected since it was average and it was considered that results obtained with it would be indicative of results obtainable with the other two bitumens. Time did not permit making complete tests with all three bitumens in this second half of the program, although spot tests were made on the other two bitumens. It was found that most of the admixtures could be reduced in quantity from the maximum recommended by the manufacturers, with a consequent improve-

TABLE V.—ANALYSIS OF MC-2 CUT-BACK BLENDED WITH THE VARIOUS ADMIXTURES.

Bitumen	Usual Specification		MC-2	MC-2	MC-2	MC-2	MC-2	MC-2
Additive			Blank	F	E	B	D	G
Additive, per cent	Minimum	Maximum	None	4	4	3	4	3
Specific gravity at 77/77 F.	...	...	0.961	0.960	0.961	0.959	0.961	...
Flash point by Tag Open Cup, deg. Fahr.	150	...	158	156	164	134[a]	134[a]	112[a]
Saybolt furol viscosity at 140 F.	100	200	139	135	136	138	140	139
Distillation, total per cent by volume of total distillate								
to 374 F.	...	...	2.1	6.0	0.0	0.0	0.0	0.0
to 437 F.	...	10	8.5	16.0[a]	9.3	8.3	7.7	17.0[a]
to 500 F.	15	55	29.8	40.0	30.2	35.4	46.2	52.8
to 600 F.	60	87	72.5	72.0	69.8	70.8	65.4	83.0
Residue from distillation to 680 F. by volume, per cent	67	...	76.5	75.0	78.5	76.0	74.0	73.5
TESTS OF RESIDUE								
Ductility at 77 F., cm.	100	...	103	109+	(1)[a,b]	(2)[a,b]	85[a]	109+
Penetration of 100 g. after 5 sec. at 77 F.	120	300	286	300+	300+[a]	300+[a]	300+[a]	265
Solubility in CCl_4, per cent	99.5	...	99.9	99.9	99.9	99.9	99.9	99.9
Oliensis spot test	Negative		Neg.	Neg.	Neg.	Neg.	Neg.	Neg.

[a] Fails specifications, as indicated.

[b] (1) Emulsifies slowly at 38. (2) Emulsifies quickly at 65.

ment in stability at no substantial sacrifice to stripping resistance.

Wetting of the aggregate particle by the bitumen is, of course, the first prerequisite in an investigation of this kind. If wetting is not substantially complete, there can be no adequate evaluation of stripping of bitumen film from the aggregate surface, or of stability of the mixture. In fact, there is no point in even considering a bituminous blend which will not coat the aggregate. Partial wetting, that is, the formation of a contact angle, denotes that the force of adhesion between the solid and the liquid (which tends to cause the liquid to spread over the surface) is less than the surface tension of the liquid, which tends to cause the liquid to gather itself into a drop. Conversely, wetting is always complete when the force of adhesion exceeds the surface tension of the liquid. The lower surface tension of bitumen over that of water sometimes makes it possible for it to coat a water-wet aggregate surface without actually adhering to it. This is termed coverage (as opposed to wetting power), and may be obtained without adhesion between the liquid and the solid. The conventional static immersion stripping test may not uncover this incipient condition and, for this reason, it was considered that some force should be applied to dislodge the film if it is susceptible to being dislodged. Hence the immersion-stability test, which is described above and which is considered more significant than a static immersion stripping test for properly evaluating the wetting power and anti-stripping properties of bitumens in bituminous concrete mixtures.

Of course, it is not known what the exact relation is between this accelerated test and performance in the field, no more than is it known what exact relation exists between accelerated laboratory freezing-and-thawing tests and durability of portland cement concrete under field weathering conditions. When it is attempted to simulate in the laboratory, by accelerated tests, such actions as field curing, saturation and soaking over a period of months or years in the prototype, and other phenomena which occur in the field, it is anybody's guess as to how accurately these simulated conditions reflect field performance. There exists a hiatus which is almost impossible to bridge without extending the program to full-scale, long range field tests. But while this laboratory test may not exactly prognosticate service behavior, it does furnish valuable comparative data on the combined effects of additives from the standpoint of relative wetting power, stripping resistance, and stability of bituminous concrete mixtures; since it simulates actual field conditions more nearly than do the conventional stripping test procedures, it is considered a more realistic procedure. Although conventional stripping tests have certain advantages, notably that of giving some indication in a relatively short time of the efficacy of an admixture (and may be quite dependable when evaluating blends for surface treatments), and in serving as preliminary trial tests in order to obtain some idea of their action before starting final tests, they also have several serious disadvantages. Generally, only one or two size fractions of an aggregate are used. This is not conducive to an accurate evaluation of wettability of an aggregate as a whole. The method of estimating stripping by visual inspection lacks precision. Correlation does not exist between results obtained by conventional stripping tests and effect of water upon a mechanical bituminous mixture. A film of bitumen may be loose on a particle of aggregate

TABLE VI.—MODIFIED OBERBACH STRIPPING TEST RESULTS.

Conditions of testing		Asphalt Cement (AC-8)								RC-2 Cut-Back								M-2 Cut-Back							
Surface of Aggregate		Dry			Wet					Dry			Wet					Dry			Wet				
Curing Temperature, deg. Fahr.		...	77	140	...	77	140	F & T[a]		...	77	140	...	77	140	77 F.+	F & T[a]	...	77	140	...	77	140	77 F.+	F & T[a]
Test Temperature in Water, 24 hr., deg. Fahr.		70	100	140	70	100	140	70	100	70	100	140	70	100	140	70	100	70	100	140	70	100	140	70	100
Aggregates	Additives, per cent	Estimated Percentage Stripped								Estimated Percentage Stripped								Estimated Percentage Stripped							
Crushed gravel	Blank—0	5	50	95	30	90	100	30	90	100	95	100	100	100	75	100	100	95	95	85	100	100	95	100	100
	Agent D—4	0	0	10	0	0	50	0	0	0	5	95	0	5	75	5	5	5	5	50	5	5	40	5	5
	Agent E—4	0	0	0	0	0	0	0	0	0	5	15	0	5	20	5	5	5	5	25	5	5	35	5	5
	Agent I—3	0	5	5	0	5	5	0	20	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
	Agent B—3	...	...	...	...	...	...	...	...	0	0	10	0	0	75	0	0	10	5	40	10	5	75	5	15
	Agent F—4	...	...	...	...	...	...	...	...	0	5	15	0	0	50	0	0	5	15	60	5	10	60	5	10
	Agent G—3	25	80	95	45	95	90	50	95	80	80	80	80	50	75	30	10	95	75	75	90	50	60	20	40
Quartzite	Blank—0	0	40	75	50	95	100	40	95	25	75	75	100	100	95	95	100	80	75	75	100	100	95	100	100
	Agent D—4	0	0	10	0	0	10	0	0	0	0	10	0	0	10	0	0	0	0	5	0	0	5	0	5
	Agent E—4	0	0	5	0	0	0	0	0	5	5	25	0	0	25	0	5	5	15	30	0	5	40	5	5
	Agent I—3	0	0	5	0	10	15	5	20	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
	Agent B—3	...	...	...	...	...	...	...	...	5	0	5	5	0	10	0	0	5	0	5	5	0	20	5	5
	Agent F—4	...	...	...	...	...	...	...	...	0	0	50	0	0	10	0	0	0	5	15	0	5	25	0	5
	Agent G—3	10	50	90	75	75	90	65	75	25	80	60	15	25	25	10	25	30	15	50	50	25	50	5	20
Pit-run gravel	Blank—0	5	30	95	20	75	100	50	90	90	75	95	100	100	100	95	100	75	75	85	100	100	95	100	100
	Agent D—4	0	0	15	0	0	15	0	0	0	0	0	0	0	0	0	0	0	5	5	0	5	5	5	5
	Agent E—4	0	0	0	0	0	5	0	0	0	5	25	0	5	25	0	5	5	10	40	5	10	50	5	10
	Agent I—3	0	0	5	15	20	30	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
	Agent B—3	...	...	...	...	...	...	...	...	0	0	0	0	0	0	0	0	5	0	25	5	5	5	5	5
	Agent F—4	...	...	...	...	...	...	...	...	0	0	25	0	0	30	0	0	5	0	10	5	10	50	5	10
	Agent G—3	10	50	90	90	90	80	80	80	50	75	90	50	50	95	5	25	35	50	75	60	25	60	15	50
Granite	Blank—0	25	95	100	20	95	100	90	100	90	95	95	100	100	100	100	100	90	95	90	100	100	95	100	100
	Agent D—4	0	0	5	0	0	5	0	0	0	0	5	0	0	5	0	0	0	0	0	0	0	0	0	0
	Agent E—4	0	0	0	0	0	0	0	0	5	5	5	0	0	5	0	0	0	0	0	0	0	5	0	5
	Agent I—3	10	25	50	0	5	15	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
	Agent B—3	...	...	...	...	...	...	...	...	0	0	5	0	0	20	0	0	10	0	40	10	0	15	5	0
	Agent F—4	...	...	...	...	...	...	...	...	0	0	5	0	0	5	0	0	0	5	40	0	15	60	5	5
	Agent G—3	40	75	75	90	90	30	90	90	40	30	25	30	5	15	0	5	30	50	50	40	0	5	5	5
Lime rock (Brooksville)	Blank—0	0	35	50	0	35	40	5	15	50	35	75	80	50	10	10	60	75	75	70	100	100	50	95	100
	Agent D—4	0	0	0	0	0	0	0	0	0	0	5	5	0	5	0	5	5	10	20	5	5	10	0	5
	Agent E—4	0	0	0	0	0	0	0	0	0	0	5	0	0	5	0	5	5	5	15	0	0	20	5	0
	Agent I—3	0	0	5	0	0	5	0	0	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
	Agent B—3	...	...	...	...	...	...	...	...	0	0	5	0	0	0	0	0	10	0	5	5	5	5	5	5
	Agent F—4	...	...	...	...	...	...	...	...	0	0	5	0	0	5	0	0	5	5	15	10	20	20	15	15
	Agent G—3	5	15	30	20	30	25	20	30	30	15	50	10	5	50	5	10	25	5	30	80	5	25	15	10

Coral (Reef)	Blank—0	5	5	25	5	15	25	...	...	50	65	25	100	95	85	...	...	75	60	85	100	100	95	...	...
	Agent D—4	5	5	20	5	5	25	...	...	5	5	10	5	5	40	...	...	5	10	25	25	25	35	...	...
	Agent E—4	5	5	15	0	0	5	...	...	5	5	10	5	10	15	...	...	10	10	25	15	10	25	...	...
	Agent I—3	0	5	20	0	5	10	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
	Agent B—3	...	...	...	...	...	...	...	...	5	5	15	75	10	15	...	...	5	5	15	25	10	25	...	...
	Agent F—4	...	...	...	...	...	...	...	...	5	5	30	75	5	30	...	...	25	20	75	75	50	20	...	...
	Agent G—3	0	5	15	0	0	5	...	...	5	5	10	5	15	15	...	...	25	25	60	30	25	75	...	...
Limestone	Blank—0	5	5	20	5	10	30	5	10	30	10	10	100	95	25	75	90	50	40	60	100	100	75	100	100
	Agent D—4	0	5	5	0	5	5	0	0	0	0	5	0	0	5	0	0	0	0	25	5	0	10	0	5
	Agent E—4	0	0	5	0	0	5	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	5
	Agent I—3	0	0	10	0	0	15	0	0	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
	Agent B—3	...	...	...	...	...	...	...	...	5	0	0	5	0	0	0	0	5	0	5	5	0	5	0	0
	Agent F—4	...	...	...	...	...	...	...	...	5	0	5	0	0	5	0	0	5	5	30	30	5	30	0	50
	Agent G—3	0	0	15	10	0	5	0	5	5	0	5	5	0	5	0	0	5	5	10	5	5	25	5	5
Lime rock (Ojus)	Blank—0	0	5	10	5	15	50	15	50	35	45	55	100	95	95	95	100	40	60	75	100	100	95	100	100
	Agent D—4	0	0	5	0	0	0	0	0	0	0	0	0	0	0	0	0	5	5	15	0	0	25	0	5
	Agent E—4	0	0	10	0	5	5	0	5	0	0	0	0	0	0	0	0	5	0	15	0	0	5	0	0
	Agent I—3	0	0	5	0	0	5	0	0	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
	Agent B—3	...	...	...	...	...	...	...	...	0	0	0	0	0	0	0	0	5	0	5	0	5	5	0	0
	Agent F—4	...	...	...	...	...	...	...	...	0	0	0	0	0	0	0	0	5	0	40	40	25	50	0	0
	Agent G—3	0	0	5	0	0	5	0	5	5	0	5	5	0	5	0	0	5	10	25	25	0	25	0	0
Fine sand	Blank—0	0	0	5	0	0	0	0	0	90	5	0	100	100	90	100	100	50	5	5	100	100	50	100	100
	Agent D—4	0	0	0	0	0	0	0	0	0	5	0	0	5	0	0	0	5	0	0	25	0	0	0	10
	Agent E—4	0	0	0	0	0	0	0	0	0	0	0	5	0	0	0	5	0	0	0	10	0	0	0	0
	Agent I—3	0	0	0	0	0	0	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...	...
	Agent B—3	...	...	...	...	...	...	...	...	0	0	0	0	0	0	10	10	0	0	0	25	0	0	0	0
	Agent F—4	...	...	...	...	...	...	...	...	5	5	0	5	10	5	5	5	5	5	0	20	0	0	0	0
	Agent G—3	0	5	0	10	20	5	...	...	5	0	0	5	10	0	5	5	5	0	0	25	0	0	0	0

[a] Two cycles of freezing and thawing before subjecting to stripping test. The cut-backs were cured for 24 hr. at 77 F. before placing in freezing chamber

and yet maintain its continuity under the usual stripping test—its lower surface tension is a factor which may make it possible to coat the surface-wet aggregate under favorable conditions without actually adhering to it (coverage).

TABLE VII.—MINIMUM PERCENTAGES OF ADDITIVES TO PROMOTE WETTING POWER OF RC-2 CUT-BACK MIXED WITH DIFFERENT TYPES OF SURFACE-WET AGGREGATES.

Modified Oberbach Stripping Test Results, in per cent, (at 100 F. water temperature) included.

Additive and stripped	Granite	Limestone	Pit-Run Gravel	Quartzite	Crushed Gravel	Lime Rock (Brooksville)	Lime Rock (Ojus)	Reef Coral
(Absorption of Aggregate, per cent)	0.5	0.5	0.7	1.1	3.0	3.0	5.7	6.2
Agent A	1.25	3.0	3.0	3.0	3.0	4.5±[a]	4.5±[a]	4.5±[a]
stripped	5	0	0	0	5	0	10	10
Agent B	1.75	1.25	3.0	3.0	3.0	4.5±[a]	4.5±[a]	4.5±[a]
stripped	5	0	0	0	0	0	10	10
Agent C	1.5	5.0±[a]	3.0	3.0	3.0	4.5±[a]	4.5±[a]	4.5±[a]
stripped	5	...	5	15	10	0	...	...
Agent D	1.75	3.0	3.0	4.0	4.0	4.5±[a]	4.5±[a]	4.5±[a]
stripped	0	5	0	0	0	10	10	10
Agent E	2.5	3.5	3.5	4.0	4.0±[a]	4.5±[a]	4.5±[a]	4.5±[a]
stripped	0	0	10	10	5	10	10	10
Agent F	2.5	3.0	4.0	4 0	4.0±[a]	4.5±[a]	4.5±[a]	4.5±[a]
stripped	0	5	0	5	5	10	10	10
Agent G	3.0	3.0	4.0±[a]	4.0±[a]	4.0±[a]	4.5±[a]	4.5±[a]	4.5±[a]
stripped	30	5	40	30	60	10	15	15
Agent H	4.5±[a]	4.5±[a]	4.5±[a]	4.5±[a]	4.5±[a]	4.5±[a]	4.5±[a]	4.5±[a]
stripped	...	...	..	..	...	...	...	...
No additive, stripped	100	95	100	100	100	50	95	95

[a] Did not coat satisfactorily (less than 90 per cent). Varying the per cent additive as much as 2 per cent in this range had no appreciable effect on wettability.

NOTE:—No reasonable amount of any of the additives would promote wetting power of AC-8 asphalt with surface-wet aggregates, nor that of RC-2 with the porous, wet lime rocks. In general, ¼ to ½ per cent less additive is required for MC-2 than for RC-2 to promote wetting power; this cut-back could be made to coat all wet aggregates with use of all admixtures, except for agent H with pit-run gravel, and the porous lime rocks, and except for agent C with calcareous aggregates.

TABLE VIII.—MINIMUM PER CENTS OF ADDITIVES TO PROMOTE ANTI-STRIPPING OF RC-2 CUT-BACK AND SURFACE-DRY AGGREGATE MIXES

Stripping tendency measured by the Modified Oberbach Test in per cent at 100 F. water temperature.

Additive and stripped	Pit-Run Gravel	Quartzite	Crushed Gravel	Granite	Limestone	Lime Rock (Brooksville)	Lime Rock (Ojus)	Reef Coral
Agent A	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
stripped	0	0	0	5	5	0	10	5
Agent B	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
stripped	5	5	10	5	5	5	5	10
Agent C	0.5	0.5	0.5	0.5	0.5	0.5	0.5+	1.5
stripped	0	5	10	0	5	5	25	5
Agent D	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.75
stripped	5	5	10	15	5	5	15	10
Agent E	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.75
stripped	0	5	10	5	5	5	5	5
Agent F	1.0	1.0	1.0	1.5	1.0	1.5	1.0	1.5
stripped	5	5	5	15	5	5	5	15
Agent H	0.5	0.5	0.5	0.5	0.5	0.5	0.5	1.0
stripped	5	5	10	10	0	5	10	15
Agent G	4.0+[a]	4.0+[a]	4.0+[a]	4.0+[a]	4.0	4.0	2.5	2.5
stripped	70	60	90	50	10	20	5	5
No additive, stripped	75	75	100	95	20	35	45	65

[a] 4 to 5 per cent of agent G would not appreciably improve resistance to stripping of the siliceous aggregates.

NOTE:—These same percentages, or slightly less, are equally effective with AC-8 asphalt. With MC-2 cut-back, the above percentages have to be increased about ¼ per cent to hold stripping action to par with that of RC-2.

It is noteworthy that, although all of the siliceous aggregates and the Brooksville lime rock are (to all intents and purposes) hydrophilic for the bitumens used, the reef coral, limestone, Ojus lime rock and Eglin sand are hydrophobic with AC-8 asphalt and part of them, when surface-dry, with RC-2 cut-back asphalt; yet, these four aggregates are definitely hydrophilic with MC-2 cut-back asphalt, and with RC-2 cut-back asphalt when they are surface-wet. From this, it would seem that the terms hydrophobic and hydrophilic are relative terms and dependent, among other things, upon the type of bitumen used and the surface condition of the aggregate.

SUMMARY

Typical results of wettability, stripping, and immersion-stability tests are given in Tables VI to XIII, inclusive. To show in detail all results obtained would require more space than is justified in this paper, especially in view of the fact that the materials are anonymous. Summarized below are results as reflected by the data obtained from the laboratory tests.

Table VI shows in detail the Oberbach stripping characteristics of admixtures tested during the initial stage of the investigation. This table represents the first series of tests made, being completed before it was fully realized that the static stripping test fell short of giving a realistic answer. It will be observed that this table gives results with maximum percentages of the different agents. Agent G is the only one of the additives which showed up poorly in this test.

Table VII shows the minimum percentages of the different agents, combined with RC-2 cut-back asphalt, required to promote wetting power for that bitumen when mixed with surface-wet aggregates. The amounts of these same agents required to lend wetting power to MC-2 cut-back asphalt range from $\frac{1}{4}$ to $\frac{1}{2}$ per cent less. No reasonable amount of any of the agents would promote wetting power of the AC-8 asphalt when mixed with surface-wet aggregates. In a few instances, notably with 33 per cent of agent D blended with AC-8 asphalt for coating wet Brooksville lime rock, wetting power of the asphalt cement was achieved; this, however, at the cost of practically all adhesion and stability of the mix. This may be due to the fact that, while low viscosity in an asphalt aids the wetting of an aggregate, high viscosity and lowered penetration are important deterrents to film stripping and low stability. None of the agents tested, used in amounts approaching 5 per cent, would promote wetting power of the cut-back asphalts when mixed with wet, porous lime rocks. The results of the modified Oberbach stripping test, as evaluated by samples tested at a water temperature of 100 F., are included in the table. All the agents, except G and H, showed good results as measured by that test. This test condition, static immersion after 24-hr. curing at 77 F., was chosen as the basis of comparison since it was considered more realistic than either the "no cure" method or the cure and immersion at 140 F.

Table VIII shows the approximate minimum percentage of each admixture for promoting anti-stripping of RC-2 cut-back asphalt from the different aggregates in a surface-dry state. Agent G was the only additive which, when used in reasonable amounts, did not seem to overcome the tendency of the bitumen to strip. MC-2 cut-back asphalt requires approximately $\frac{1}{4}$ per cent more admixture than does RC-2 cut-back asphalt in order to perform in the same manner. AC-8 asphalt requires about the same amount of admixture as does RC-2 cut-back asphalt.

Tables IX through XIII show typical results of the axial-compression stability tests for surface-wet and surface-dry aggregates, both for soaked and unsoaked specimens. The entire set of tabular results, 16 tables, is too volumi-

TABLE IX.—SUMMARY OF AXIAL COMPRESSION STABILITY TESTS ON SOAKED AND UNSOAKED SPECIMENS (GRANITE)

	Additive	Blank (0%)		Agent D (4%)		Agent E (4%)		Agent B (3%)		Agent F (4%)		Agent G (3%)		Agent H (4.5%)	
	Aggregate Surface	Dry		Wet		Wet		Wet		Wet		Wet		Wet	
	Saturation and Soaking	None	24-hr.	None	24-hr.	None	24-hr.	None	24-hr.	None	24-hr.	None	24-hr.	None	24-hr.
RC-2	Wettability, per cent coated	100		100		100		100		100		90		65	
	Absorption, per cent	...	6.4	...	4.2	...	3.5	...	4.3	...	4.1	...	6.0		
	Stability, lb	1185	425	573	530	578	697	904	879	820	716	1190	311		
	Stripping tendency, loss of strength of soaked specimens, per cent	0	64	0	8	0	0	0	3	0	13	0	74		
	Stability retained, based on dry blank strength, per cent														
	Soaked		100		125		163		207		168		73		
	Unsoaked	100		48		49		77		69		100			
MC-2	Wettability, per cent coated	100		100		100		100		100		90			
	Absorption, per cent	...	5.3	...	3.1	...	2.6	...	3.8	...	3.6	...	...		
	Stability, lb.	267	40	173	143	105	109	242	222	198	158	405	0		
	Stripping tendency, loss of strength of soaked specimens, per cent	0	85[a]	0	18	0	0	0	8	0	20	0	100[b]		
	Stability retained, based on dry blank strength, per cent														
	Soaked		100		357		272		555		395		0		
	Unsoaked	100		65		39		91		74		152			

	Additive	Blank (0%)		Agent D (1.75%)		Agent E (2.5%)		Agent B (1.75%) (1.25 for MC-2)		Agent F (2.5%)		Agent A (1.25%)		Agent C (1.5%)	
	Aggregate Surface	Dry		Wet		Wet		Wet		Wet		Wet		Wet	
	Saturation and Soaking	None	48-hr.	None	48-hr.	None	48-hr.	None	48-hr.	None	48-hr.	None	48-hr.	None	48-hr.
RC-2	Wettability, per cent coated	100		95		100		100		98		100		100	
	Absorption, per cent	...	6.3	...	4.8	...	3.8	...	4.7	...	4.8	...	4.3	...	5.5
	Stability, lb.	1002	366	1156	780	840	850	919	672	1062	765	830	707	835	672
	Stripping tendency, loss of strength of soaked specimens, per cent	0	63	0	33	0	0	0	27	0	26	0	15	0	20
	Stability retained, based on dry blank strength, per cent														
	Soaked		100		213		232		183		209		193		183
	Unsoaked	100		115		84		92		106		83		83	
MC-2	Wettability, per cent coated	100						100							
	Absorption, per cent	...	6.0					...	4.5						
	Stability, lb.	474	104					370	260						
	Stripping tendency, loss of strength of soaked specimens, per cent	0	78					0	28						
	Stability retained, based on dry blank strength, per cent														
	Soaked		100						256						
	Unsoaked	100						78							

[a] Started to crack.
[b] Disintegrated.

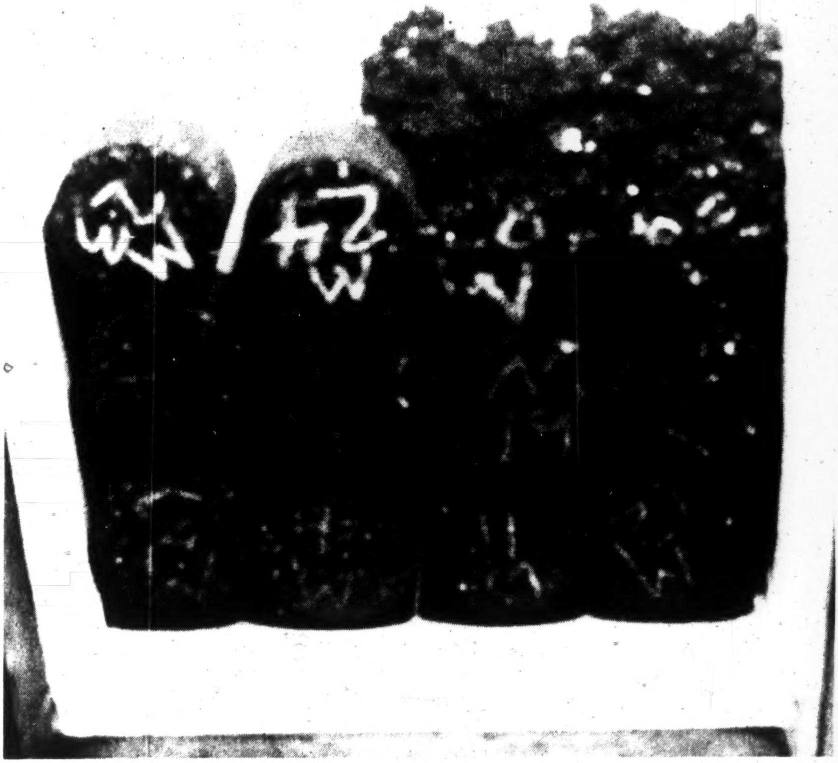

FIG. 8.—MC-2 and Granite Mixes in Water Bath. Note the disintegration of the two specimens treated with agent G.

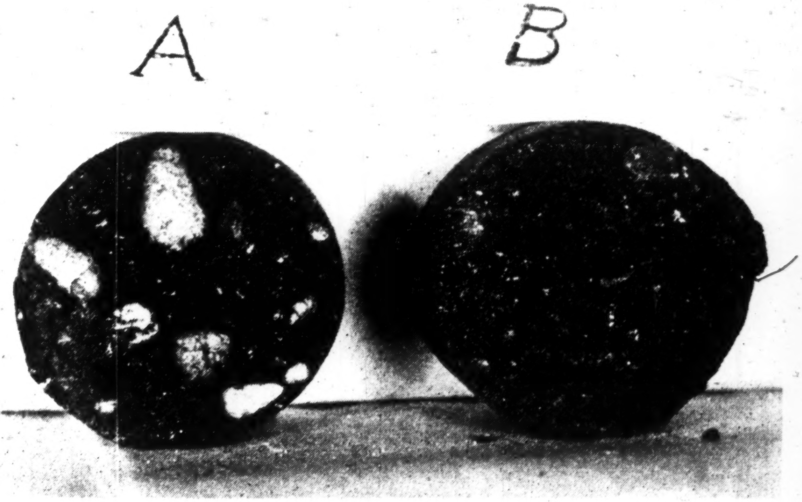

FIG. 9.—AC-8 and Pit-Run Gravel Mix.

A is without admixture. Note almost complete stripping of coarse aggregate after 24-hr. soaking.
B is treated with agent I. This view is typical of the more effective admixtures.

TABLE X.—SUMMARY OF AXIAL COMPRESSION STABILITY TESTS ON SOAKED AND UNSOAKED SPECIMENS (PIT-RUN GRAVEL).

	Additive	Blank (0%)		Agent D (4%)		Agent E (4%)		Agent B (3%)		Agent F (4%)		Agent G (4%)[a]		Agent H (4.5%)	
	Aggregate Surface	Dry		Wet		Wet		Wet		Wet		Wet		Wet	
	Saturation and Soaking	None	24-hr.	None	24-hr.	None	24-hr.	None	24-hr.	None	24-hr.	None	24-hr.	None	24-hr.
RC-2	Wettability, per cent coated	100		95		100		100		100		75		75	
	Absorption, per cent	...	6.4	...	5.4	...	6.4	...	6.5	...	6.5	...	7.0		
	Stability, lb.	306	202	54	69	79	139	178	237	168	227	308	0		
	Stripping tendency, loss of strength of soaked specimens, per cent	0	34	0	0	0	0	0	0	0	0	0	100		
	Stability retained, based on dry blank strength, per cent														
	Soaked		100		34		69		118		113		0		
	Unsoaked	100		18		26		58		55		100			
MC-2	Wettability, per cent coated	100		100				100		98					
	Absorption, per cent	...	5.7	...	5.2			...	5.0	...	5.1				
	Stability, lb.	120	79	79	109			54	59	75	110				
	Stripping tendency, loss of strength of soaked specimens, per cent	0	34	0	0			0	0	0	0				
	Stability retained, based on dry blank strength, per cent														
	Soaked		100		138				75		138				
	Unsoaked	100		66				45		65					
	Additive	Blank (0%)		Agent D (3%)		Agent E (3.5%)		Agent B (3%)		Agent F (4%)		Agent A (3%)		Agent C (3%)	
	Aggregate Surface	Dry		Wet		Wet		Wet		Wet		Wet		Wet	
	Saturation and Soaking	None	48-hr.	None	48-hr.	None	48-hr.	None	48-hr.	None	48-hr.	None	48-hr.	None	48-hr.
RC-2	Wettability, per cent coated	100		100		95		100		95		100		100	
	Absorption, per cent	...	6.3	...	5.3	...	6.5	...	5.8	...	6.3	...	6.8	...	...
	Stability, lb.	173	112	74	94	74	84	64	94	64	163	49	49	50	0
	Stripping tendency, loss of strength of soaked specimens, per cent	0	35	0	0	0	0	0	0	0	0	0	0	0	100
	Stability retained, based on dry blank strength, per cent														
	Soaked		100		84		75		84		146		44		0
	Unsoaked	100		43		43		37		37		28		29	
RC-2	Soaked		26 days		26 days		26 days		26 days		26 days		26 days		26 days
	Absorption, per cent	...	6.5	...	5.4	...	6.7	...	6.0	...	6.3	...	...	...	...
	Stability, lb.		138		99		49		59		168		0		0
	Stripping tendency, loss of strength of soaked specimens, per cent		20		0		0		0		0		100[b]		100[a]
	Stability retained, based on dry blank strength, per cent		100		72		36		43		122		0		0

[a] Disintegrated in water bath at from 4 to 16 hr.
[b] Disintegrated in water bath on 9th day

TABLE XI. SUMMARY OF AXIAL COMPRESSION STABILITY TESTS ON SOAKED AND UNSOAKED SPECIMENS (PIT RUN GRAVEL).

	Additive	Blank (0%)		Agent D (4%)		Agent E (4%)		Agent I (3%)[a] Agent B (3%)		Agent F (4%)		Agent G (3%)					
	Aggregate Surface	Dry		Dry		Dry		Dry		Dry		Dry					
	Saturation and Soaking	None	24-hr.	None	24-hr.	None	24-hr.	None	24-hr.	None	24 hr.	None	24-hr.	None	24-hr.	None	24 hr.
AC-8	Wettability, per cent coated	100		100				100				100					
	Absorption, per cent	...	5.7		5.3			...	5.4			...	6.0				
	Stability, lb.	666	435	420	602			740	1052			665	260				
	Stripping tendency, loss of strength of soaked specimens, per cent	0	34	0	0			0	0			0	61				
	Stability retained, based on dry blank strength, per cent																
	Soaked		100		138				242				60				
	Unsoaked	100		63				111				100					

	Additive	Blank (0%)		Agent D (0.5%)		Agent E (0.5%)		Agent I (0.5%)[a] Agent B (0.5%)		Agent F (1.5%)		Agent A (0.5%)		Agent C (0.5%)		Agent H (0.5%)	
	Aggregate Surface	Dry		Dry		Dry		Dry		Dry		Dry		Dry		Dry	
	Saturation and Soaking	None	48-hr.	None	48-hr.	None	48 hr.	None	48 hr.	None	48-hr.	None	48-hr.	None	48-hr.	None	48-hr.
RC-2	Wettability, per cent coated	100		100		100		100		100		100		100		100	
	Absorption, per cent	...	6.0	...	6.6	...	6.5	...	6.9	...	6.5	...	6.6	...	6.8	...	7.3
	Stability, lb	330	286	286	459	198	405	217	449	114	242	232	435	232	430	331	524
	Stripping tendency, loss of strength of soaked specimens, per cent	0	13	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	Stability retained, based on dry blank strength, per cent																
	Soaked		100		160		141		155		85		152		150		183
	Unsoaked	100		87		60		66		35		70		70		100	
RC-2	Soaked		12 days		12 days		12 days		12 days		12 days		12 days		12 days		12 days
	Absorption, per cent		6.5		6.7		6.4	...	7.0		6.8	...	6.6	...	7.0	...	7.1
	Stability, lb.		251		396		380		385		232		405		360		513
	Stripping tendency, loss of strength of soaked specimens, per cent		24		0		0		0		0		0		0		0
	Stability retained, based on dry blank strength, per cent		100		158		152		153		93		162		144		202
MC-2	Wettability, per cent coated	100						100									
	Absorption, per cent	...	6.2					...	6.4								
	Stability, lb.	333	110					165	115								
	Stripping tendency, loss of strength of soaked specimens, per cent	0	67					0	30								
	Stability retained, based on dry blank strength, per cent																
	Soaked		100						101								
	Unsoaked	100						50									

[a] Agent I for asphalt cement and agent B for cut-backs.

TABLE XII.—SUMMARY OF AXIAL COMPRESSION STABILITY TESTS ON SOAKED AND UNSOAKED SPECIMENS (BROOKSVILLE LIME ROCK—INDICATIVE OF OJUS LIME ROCK AND REEF CORAL).

	Additive	Blank (0%)		Agent D (4%)		Agent E (4%)		Agent I (3%)[a] Agent B (3%)		Agent F (4%)		Agent G (3%)					
	Aggregate Surface	Dry		Dry		Dry		Dry		Dry		Dry		Dry		Dry	
	Saturation and Soaking	None	24-hr.	None	24-hr.	None	24-hr.	None	24-hr.	None	24-hr.	None	24-hr.	None	24-hr.	None	24-hr.
AC-8	Wettability, per cent coated	100		100		100		100				100					
	Absorption, per cent	...	3.5	...	4.0	...	3.3	...	4.2			...	6.3				
	Stability, lb.	1748	967	1007	765	1017	854	1096	790			636	158				
	Stripping tendency, loss of strength of soaked specimens, per cent	0	45	0	24	0	17	0	28			0	75				
	Stability retained, based on dry blank strength, per cent																
	Soaked		100		79		88		82				16				
	Unsoaked	100		58		59		63				36					

	Additive	Blank (0%)		Agent D (0.5%)		Agent E (0.5%)		Agent I (0.5%)[a] Agent B (0.5%)		Agent F (1.5%)		Agent A (0.5%)		Agent C (0.5%)		Agent H (0.5%)	
	Aggregate Surface	Dry		Dry		Dry		Dry		Dry		Dry		Dry		Dry	
	Saturation and Soaking	None	48-hr.	None	48-hr.	None	48-hr.	None	48-hr.	None	48-hr.	None	48-hr.	None	48-hr.	None	48-hr.
AC-8	Wettability, per cent coated	100		100		100		100				100		100		100	
	Absorption, per cent	...	4.6	...	4.6	...	4.9	...	4.6			...	5.0	...	5.5	...	5.2
	Stability, lb.	1738	710	1886	973	1911	1152	2202	1052			1886	1081	1802	1017	1827	820
	Stripping tendency, loss of strength of soaked specimens, per cent	0	59	0	48	0	39	0	52			0	43	0	45	0	55
	Stability retained, based on dry blank strength, per cent																
	Soaked		100		137		162		148				152		143		115
	Unsoaked	100		109		110		127				109		106		105	
RC-2	Wettability, per cent coated	100		100		100		100		100		100		100		100	
	Absorption, per cent	...	5.4	...	4.9	...	5.2	...	5.5	...	4.9	...	4.9	...	5.5	...	5.0
	Stability, lb.	1728	628	1921	760	1975	780	2310	1063	2133	992	2088	765	1920	647	1867	696
	Stripping tendency, loss of strength of soaked specimens, per cent	0	64	0	60	0	60	0	54	0	53	0	63	0	66	0	63
	Stability retained, based on dry blank strength, per cent																
	Soaked		100		121		124		169		157		121		103		111
	Unsoaked	100		111		114		134		123		121		111		108	
MC-2	Wettability, per cent coated	100						100				100					
	Absorption, per cent	...	5.1					...	4.7			...	4.9				
	Stability, lb.	1000	250					1400	690			900	375				
	Stripping tendency, loss of strength of soaked specimens, per cent	0	75					0	51			0	58				
	Stability retained, based on dry blank strength, per cent																
	Soaked		100						275				150				
	Unsoaked	100						140				90					

[a] Agent I for asphalt cement and Agent B for cut-backs.

TABLE XIII. SUMMARY OF AXIAL COMPRESSION STABILITY TESTS ON SOAKED AND UNSOAKED SPECIMENS.

QUARTZITE

	Additive	Blank (0%)		Agent D (4.0%)		Agent E (4.0%)		Agent B (3.0%)		Agent F (4.0%)		Agent A (3.0%)		Agent C (3.0%)	
	Aggregate Surface	Dry		Wet		Wet		Wet		Wet		Wet		Wet	
	Saturation and Soaking	None	48-hr.	None	48-hr.	None	48-hr.	None	48-hr.	None	48-hr.	None	48-hr.	None	48-hr.
RC-2	Wettability, per cent coated	100		98		100		98		95		98		100	
	Absorption, per cent		6.0	...	4.7	...	4.8		5.3	...	5.6	...	4.8		5.4
	Stability, lb.	1131	778	647	785	1200	1525	889	1145	1490	1745	746	1230	1299	1471
	Stripping tendency, loss of strength of soaked specimens, per cent	0	32	0	0	0	0	0	0	0	0	0	0	0	0
	Stability retained, based on dry blank strength, per cent														
	Soaked		100		101		197		147		225		158		179
	Unsoaked	100		57		106		79		132		66		115	
RC-2	Soaked, per cent		60 days		60 days		60 days		60 days		60 days		60 days		60 days
	Absorption, per cent		7.7		5.6		5.7		6.7		6.5		6.2		6.8
	Stability, lb.		657		756		1475		879		1680		978		1220
	Stripping tendency, loss of strength of soaked specimens, per cent		42	0	0	0	0	0	1	0	0	0	0	0	6
	Stability retained, based on dry blank strength, per cent		100		115		225		134		256		149		185

GRANITE

	Additive	Blank (0%)		Agent D (1.75%)		Agent E (2.5%)		Agent B (1.75%)		Agent F (2.5%)		Agent A (1.25%)		Agent C (1.5%)	
	Aggregate Surface	Dry		Wet		Wet		Wet		Wet		Wet		Wet	
	Saturation and Soaking	None	48-hr.	None	48-hr.	None	48-hr.	None	48-hr.	None	48-hr.	None	48-hr.	None	48-hr.
RC-2	Wettability, per cent coated	100		98		100		100		98		100		100	
	Absorption, per cent		6.9		5.2	...	4.2		4.4	...	5.0		3.9	...	5.5
	Stability, lb.	750	232	904	800	903	978	790	795	894	711	760	706	620	425
	Stripping tendency, loss of strength of soaked specimens, per cent	0	69	0	11	0	0	0	0	0	20	0	7	0	32
	Stability retained, based on dry blank strength, per cent														
	Soaked		100		344		422		342		306		304		183
	Unsoaked	100		121		121		105		119		101		83	
RC-2	Soaked		60 days		60 days		60 days		60 days		60 days		60 days		60 days
	Absorption, per cent		9.3+		6.6		5.4		5.6		6.4		5.3		6.7
	Stability, lb.		64[a]		642		825		726		651		675		405
	Stripping tendency, loss of strength of soaked specimens, per cent		91		29		9		8		27		11		35
	Stability retained, based on dry blank strength, per cent		100		1002		1297		1132		1016		1053		633

[a] One specimen disintegrated at about 43 + days and the other broke at 128 lb.

nous to include in this paper. However, any reader interested in the results in greater detail may obtain copies of the other tables from the author. Figure 8 shows a view of one group of specimens in the water bath, one set of which had disintegrated; and Fig. 9, a typical section of treated and untreated companion specimens after they had been broken in the compression machine.

Another phenomenon encountered was that those agents which promoted greatest wetting power, or which had to be used in relatively large amounts in order to promote wetting power of the bitumen, seem to slow up the curing process of the bitumen in bituminous mixtures. This may be due to some inhibitory effect of the admixture upon the original physical characteristics of the bitumen or it may be that surface moisture on the aggregate is entrapped more completely, thus slowing up the curing process. In any event, it appears that the element of time is an important factor in developing maximum adhesion between an aggregate and a bitumen. It follows that a fresh asphalt film may not develop sufficient adhesion to an aggregate to prevent it from stripping in the presence of water, whereas if protected from the action of water for a sufficient length of time it may become highly resistant to stripping.

Comments not generalized above, and covering specific points of each additive, follow:

Agent A.—This was one of the most effective agents tested, in spite of the fact that the quantity used is many times that recommended by the manufacturer (the manufacturer advised that this additive, and agents C and H, would be effective when used in the amount of 0.03 to 0.07 per cent). Stripping resistance as measured by the modified Oberbach test was good. In general, the stability of mixtures in which this agent was used (in amounts ranging from 0.5 per cent with surface-dry aggregates to 4.5 per cent with surface-wet porous lime rocks) was satisfactory, except with the wet pit-run gravel and, of course, with the wet, porous lime rocks. The relatively high penetration of the blend (Table IV), plus the fact that the pit-run gravel was rounded, probably accounts for the low stability of that mix. Mixes in which a 3 per cent blend was used with the other siliceous materials, and with the limestone, were satisfactory.

Agent B.—In general, comments made in the above paragraph apply to this additive. It is equally, if not slightly more, effective than is agent A, especially with the surface-wet aggregates.

Agent C.—This agent, used with the bitumens tested, would not lend wettability to any of the surface-wet calcareous aggregates, nor was it effective from the standpoint of stability with wet pit-run gravel. It was effective, however, in promoting anti-stripping of dry aggregates, and was effective from the standpoint of wetting power, stability, and anti-stripping for the surface-wet siliceous aggregates, except pit-run gravel.

Agent D.—This agent was quite satisfactory for promoting anti-stripping when used with the dry aggregates; and with all surface-wet siliceous aggregates except pit-run gravel, which failed in stability. Increased penetration of the blend, plus the fact that pit-run gravel is rounded, may account for this.

Agent E.—Comments made for agent D apply generally to this agent, except that it had to be used in larger quantities with the surface-wet aggregates in order to promote wetting power.

Agent F.—The amount of this agent had to be increased all along the line to promote wetting power and stripping resistance of the bitumens. It was the only additive which was effective with surface-wet pit-run gravel from the standpoint of stability, in spite of the fact that it increases penetration of the bitumen. It was relatively inefficient in lending wettability to the crushed gravel. Otherwise, this agent is about on a par with agent D.

Agent G.—This additive proved generally unsatisfactory in most respects with most of the aggregates. When used in amounts approaching 5 per cent it would not lend wettability to many of the surface-wet aggregates; nor promote stripping resistance when used with siliceous aggregates. Results with MC-2 cut-back asphalt indicate that if the agent were blended with less viscous cut-back asphalts it might lend wettability to certain surface-wet aggregates, not obtainable with the more viscous bitumens.

Agent H.—This agent is effective with the surface-dry aggregates. When used in amounts approaching 5 per cent it will not promote wettability of any of the surface-wet aggregates. For this reason it could not be evaluated from the standpoint of stripping tendency and stability of the wet aggregate mixes.

Agent I.—This agent is used only in conjunction with asphalt cements. The most satisfactory approximate amount for use with all the aggregates except porous lime rocks which require some 2 to 3 per cent of the additive for best performances is 0.5 per cent with AC-8 asphalt. In common with other additives, it will not promote the wetting power of the asphalt cement (AC-8) for coating surface-wet aggregates.

Accelerated freezing-and-thawing tests, conducted at two cycles and spot checked at 25 cycles, did not show any significant effect on the stripping properties of the admixtures.

Tables X, XI and XIII contain information on specimens soaked in water, respectively, for 26, 12, and 60 days. Table XI shows no appreciable difference in results between 48-hr. and 12-day soaking periods. Table XIII is of considerable interest in that it shows a comparison of results for companion specimens soaked, respectively, 48 hr. and 60 days. It shows that, although the blank mixes tend to deteriorate rapidly with time when subjected to moisture, there is considerable benefit to be derived from the use of certain admixtures under similar conditions, in that this deterioration is arrested, or at least slowed down. This is particularly remarkable with an aggregate having the characteristics of the granite used in these tests—although the blank mix with this aggregate disintegrated, the blended mixes with the more effective admixtures held up well for the period of test. Whether they would hold up as well for 600 days, or 6000 days, is a moot question which could be answered only by making actual long-range field tests. But when it is considered that 60 days of continuous soaking may well simulate many times that age of normal field moisture conditions of alternate wetting and drying, the test offers encouragement. The results indicate that the accelerated soaking test has considerable merit in pointing the way and making a laboratory evaluation of an admixture which is being considered for use as a wetting and/or anti-stripping agent.

CONCLUSIONS

Within the limits of the tests reported herein, and keeping in mind that no known correlation has been established between field performance and accelerated laboratory tests on this type of construction material, it is concluded that:

1. Conventional static immersion stripping tests, *per se*, not only do not tell the full story for plant-mixed bitu-

minous concrete but may even lead (if used alone) to a favorable report on an anti-stripping admixture when, as a matter of fact, this agent might do more harm than good in a bituminous concrete mix. The use of a selected additive for a given type of aggregate without sufficient preliminary examination of the finished mixtures may produce unsatisfactory results.

2. None of the additives tested, when used in amounts optimum for stability and stripping resistance, will promote wetting power of asphalt cement in the consistency range covered, for coating of surface-wet aggregate; nor the wetting power of the cut-back asphalts tested for coating the wet, porous lime rocks. (Indications are that some of the agents will promote the wetting power of lighter bitumens, to the end that they might satisfactorily coat most types of surface-wet aggregates.)

3. When used in conjunction with surface-dry aggregates, all of the agents (except agent G with siliceous aggregates) promote wetting power, stripping resistance, and stability of bituminous mixtures.

4. In general, when used in conjunction with surface-wet aggregates, agents A, B, and C are the most effective of the additives tested, subject to the limitations noted in (3) above. Agents D and F are only slightly less effective, the latter being the only additive which was effective with the wet pit-run gravel. Agents G and H are the least effective additives. It is very evident that, in some cases, the satisfactory use of an additive is limited to certain types of aggregates and under certain conditions.

5. Relatively high percentages of additives added to bitumen as materials may have detrimental effects on their physical characteristics.

6. The terms "hydrophilic" and "hydrophobic" should not be associated unreservedly to a given aggregate for all conditions. The results suggest that an aggregate might be hydrophilic to a given grade of bitumen, yet be hydrophobic to another, or it might be hydrophilic, when surface-wet, to a bitumen—yet be hydrophobic when used in a dry state with that same bitumen.

7. Further research work by the manufacturers is in order to increase the efficiency of additives and develop more definite methods of use under varying conditions.

NOTE.—While not proven in this investigation, results of laboratory tests indicate that those additives which promoted wetting power and stripping resistance could be expected to be beneficial in penetration-type bituminous construction.

The thought is offered that, until such time as some dependable method has been widely accepted and standardized for general use, the axial-compression strength stability test on soaked as compared with unsoaked specimens may be useful as a wetting power and stripping tendency laboratory test for evaluating admixtures proposed for use in bituminous plant mixtures; also, that it be viewed in the light of a quasi field test, stipulating that long-time soaking tests be conducted in the laboratory as a check on accelerated soaking tests. The merit in using this as a quasi field test is that it can save much large-scale field investigation work, and should reveal as much, if not more, information than abbreviated field investigations. The conventional stripping test could be used as an indicative trial test for obtaining information of a preliminary nature for application to the immersion-stability test, and as an indication with reference to penetration-type construction.

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der the general direction of Brigadier Generals R. F. Fowler and James B. Newman, Jr., successively Division Engineers, South Atlantic Division, Corps of Engineers, U. S. Army; and of R. W. Stuck, Chief Civilian Engineer, South Atlantic Division. The author wishes to make special mention of the valuable suggestions given by the staff of the Engineer Board, Fort Belvoir, Va.; and of the keen spirit and untiring loyalty which the staff of the Division Laboratory exhibited in compiling and analyzing the test data.

Acknowledgment is made to the staff of the Public Roads Administration, whose helpful and valuable suggestions led directly to the use of the immersion-stability method for evaluating admixtures in plant-type bituminous concrete.

APPENDIX

Description of Axial-Compression Stability Test

The first step in developing this phase of the program was to establish criteria for accelerated testing which would be as realistic as practicable in so far as simulating actual field conditions is concerned, yet requires a minimum of time for laboratory tests. Among the problems to be solved were method and time of mixing, maximum size and grading of aggregate, size and minimum number of specimens for each condition, time and condition of preliminary curing, saturating and soaking of "soaked" specimens, and proper interpretation of results obtained.

Because of the relatively large size of the laboratory batches, and in order to simulate field conditions as nearly as practicable, it was decided to construct and use a small laboratory single pugmill. Nominal mixing time was established at 2 min., although it was found necessary to vary the time somewhat in order to produce the maximum obtainable degree of coating.

The maximum size aggregate was established at ¾ in., but it was not feasible to use a conventional job mix formula for reasons brought out below. The grading of aggregate finally decided upon is given in Table I. Each aggregate was screened into the individual particle sizes indicated, and subsequently recombined into the synthetic grading shown in the table. This assured a constant gradation throughout and, while not meeting criteria for job mix formulas for the different aggregates, lends itself more readily to uniformity and reproducibility of results for the type tests contemplated.

Cylindrical specimens 3 in. in diameter and 3 in. high were decided upon as being satisfactory from standpoint of ratio of aggregate size to size of specimen, and of conserving time and material in molding and testing. After a few trial tests it was decided that mixes should not be molded under a pressure exceeding 2000 psi. because of danger of degradation of some of the softer aggregates. Consequently, all specimens were compressed by double-plunger (freely moving pistons at both ends of the mold) at 2000 psi. The double-plunger method assured a substantially uniform density throughout the specimen. Preliminary tests indicated sufficient uniformity and reproducibility as to warrant making only four specimens to a set.

Another expedient resorted to in order to speed up laboratory tests was to accelerate the curing of the mixtures. As the result of a limited number of preliminary trials it was decided to cure the cut-backs in a forced-draft oven at 140 F., allowing 24 hr. for the RC-2 cut-back asphalt and 48 hr. for the MC-2 cut-back asphalt. The mixtures were cured in a loose state, and allowed to cool to room temperature before molding. AC-8 asphalt required no preliminary curing and was molded, while hot, immediately after mixing.

The most difficult problem was that of trying to establish a suitable method for obtaining substantially complete saturation in a very short period of time so that a nominal soaking period (of the order of 24 to 48 hr.) could be obtained without delay. It was a foregone conclusion that a mixture which was tight enough to have measurable strength under the worst conditions would take days, and perhaps weeks, of continuous immersion at atmospheric pressure in water to become sufficiently saturated so that effective soaking could begin to take effect. On the other hand, it was feared that a condition of vacuum high enough to accelerate saturation of tight specimens might at the same time have an adverse effect upon their structural properties. The problem, therefore, was to strike a proper balance between density of mix and a vacuum which was sufficiently low so that it had no appreciable adverse effect upon the structure of the specimen.

The problem was simplified somewhat since relative, not absolute, values were required. AC-8 asphalt was used with dry Brooksville lime rock for the preliminary trial tests. A 97 per cent density mix was obtained with a typical surface-course job mix formula. A 92 per cent density mix was produced by a straight line grading from ¾ in. to No. 40 aggregate. Gradation for a 95 per cent density mix followed the typical job mix formula curve down to No. 10 size, and assumed a straight line grading from there to zero per cent passing the No. 80 sieve. It was the latter gradation which was finally decided upon. It was found that the tight mix immersed in water at atmospheric pressure was not affected appreciably even at the end of 9 days, and that action at that stage was decelerating. On the other hand, although relatively quick action was possible with the 92 per cent density mix, its strength range was considered too low for measurable results of stability where cut-back asphalts would be mixed with surface-wet aggregate. Further, even with this low density, complete saturation was not possible under several days, unless the mix was first subjected to a vacuum.

As a result of the relative effects of a 76-cm. vacuum, for periods ranging up to an hour, on the tight and loose mixes, it was decided to try modified vacuums for short periods on a 95 per cent density mix. The high vacuum on the tight mix might have proved satisfactory were it not for the fact that in the process of evacuating air from the specimen it also pulled water from around the saturated and surface-wet aggregate particles, and ruptured the bituminous film in the process. On the other hand, a vacuum as low as 15 cm. removed only a negligible amount of air from the specimens. In the final analysis, with the limited amount of time which was available for this one research problem, it was decided that fairly satisfactory results would be obtained by designing the mix for a density of 95 per cent, subjecting specimens which were to be soaked to a 30-cm. vacuum for 15 min. and immersing them in a 77 F. water bath for a 24-hr. soaking period. (The 24-hr. soaking period was later increased to 48 hr.) While this may not be the optimum condition, it does permit almost instantaneous saturation, which in turn makes it possible to obtain soaking in a minimum of time.

Interpretation of test results involved a comparison between soaked and unsoaked specimens (considered a measure of stripping tendency) within a family group, and between family groups; each family group (all specimens for a condition of test) was in turn evaluated in terms of results obtaibed with the blank family group. End results shown in the "stability" Tables IX through XIII were determined as follows:

Stripping Tendency—per cent Loss of Strength of Soaked Speciemns:

The arithmetical difference between the numerical stability values of unsoaked and soaked specimens of a family group is divided by the numerical stability values of unsoaked specimens, the result being percentage loss in stability resulting from the soaking action of water. This is considered a "stripping tendency" although it is not necessarily an actual measure of stripping, *per se.* It is possible that factors other than stripping are exerting an influence on the stability results.

Example: Unsoaked stability = 267 lb.
Soaked stability = 40 lb.

$$\text{Stripping Tendency} = \frac{267-40}{267} = 85 \text{ per cent}$$

Thus, in this particular instance (which happens to be a plain bituminous mix of MC-2 cut-back and surface-dry granite) the stripping tendency is 85 per cent. This is in general accord with the modified Oberbach stripping result for these same materials—generally speaking, this accord obtains throughout with most of the combinations.

Per cent Stability Retained, Based on Dry Blank Strength (Soaked, Unsoaked):

Of course, "dry blank strength" refers

to the axial-compression stability of mixtures of bitumens (without admixtures) and surface-dry aggregates. Unsoaked and soaked stability values of specimens in this family group are divided, respectively, into unsoaked and soaked stability values of specimens in other family groups, the end result being the ratio of stability of blended specimens to stability of blank specimens, expressed on a percentage basis.

Example:

Stability	Blank		Blend	
	Unsoaked	Soaked	Unsoaked	Soaked
	267	40	173	143
Stability retained, per cent				
Soaked.		100		375
Unsoaked.	100		65	

(This example involves the same blank mixture which showed a stripping tendency of 85 per cent, above. The companion blend, using 4 per cent of agent D, showed a stripping tendency of 18 per cent.)

While the stripping tendency, as such, may not be an actual measure of stripping in the commonly accepted sense of the word, it certainly shows clearly any tendency toward strength or weakness in the structure of a mixture, induced by actual stripping action and perhaps by other factors (such as initial adhesion induced by wetting power). Among other things it tends to seek out those incipient, or potential stripping cases where the film of bitumen adheres but loosely to an aggregate particle and which is not detectable under ordinary conventional stripping tests.

As can be expected, there exists a general relationship between penetration of the bitumens (see Physical Characteristics, Tables III, IV, and V) and stability of the unsoaked specimens. Increased penetration of the bitumen, everything else being equal, tends to reflect in lowered stability of the mixture in which the bitumen is used.

The stability of the soaked specimens is believed to be a function of opposing influences, major among them being penetration and viscosity of the bitumen *versus* tendency of the bitumen film to strip (or not to strip) from the aggregate particles. In other words, since the blank, or plain bituminous mix, is established as a datum with which all other mixes (blends of bitumens and admixtures) are compared, in the final analysis they should have equal or better stabilities than the datum in order to be acceptable for use. If an admixture, because of increased penetration or lowered viscosity induced in the bitumen blend by its use, produces mixtures with substantially lower stability than the plain bituminous mix itself, then it has questionable value. Of course, if it shows superior performance on specimens tested in a soaked condition, and such a condition is expected in the prototype, then any slight tendency toward lowered stability in the unsoaked specimen might well be ignored. Laboratory tests, unsupported by long-range field tests, can go only so far in developing this point. A relatively short-range laboratory test might show the stability of a bitumen-admixture blend as being lower than that of the bituminous mix itself; yet over a period of months or years the blend, by virtue of its greater resistance to stripping action might well prove its worth. On the other hand, the fact that there was no appreciable difference in results between 48-hr. soaking and 60-day soaking (Table XIII) is a good indication that the accelerated immersion-stability test, if used and interpreted correctly, can be reasonably dependable in most cases in prognosticating fairly accurately field performance of anti-stripping admixtures.

DISCUSSION

MR. A. HOLMES[1] (*by letter*).—Mr. Sprague's investigation on the effect of additives in asphalts has yielded interesting results on a currently very important subject. In reviewing the information presented there are several items which merit some elaboration. The incorporation of the additive changed the physical properties of the asphaltic product to such an extent as to cause it to fail to meet specifications. This illustrates the danger of adding the antistripping agent in mixing plants without making allowances for such changes. This is important since the stabilities of dry-surfaced aggregates depend upon the consistency of the asphalt binder. This observation emphasizes the desirability of having the antistripping agent incorporated at the refinery whereby a product meeting specifications can be assured.

In addition to absorption, surface texture, and predominating minerals, adsorption undoubtedly plays an important, if not the deciding, action in the wetting of the aggregate by the asphaltic material and the retention of the asphaltic film in the presence of water. The terms "hydrophilic" and "hydrophobic" are quite confusing when considering the aggregate in a wet or dry state. Actually all aggregates normally encountered appear to be wetted by water with equal ease. One concept that may be advanced to explain the difference between the "hydrophilic" and "hydrophobic" aggregate surfaces is that water enters into direct combination with the "hydrophilic" minerals to form hydrated compounds on the surface. It has been shown in our laboratory that the interfacial tension between asphalt and a hydrated (wetted) mineral surface is different from that for the dry aggregate surface. A "hydrophobic" aggregate is one which does not combine chemically with water although it may be easily wetted by it.

The modified Oberbach stripping tests (static immersion) were conducted under a variety of conditions: (*a*) immersion of the coated aggregate immediately after mixing without allowing any curing; (*b*) curing at 77 F. for 24 hr. followed by immersion at 100 F.; (*c*) curing at 140 F. for 24 hr. and then immersed in water at 140 F.; (*d*) curing at 77 F. followed by freezing-and-thawing cycles before immersion in water at 70 and 100 F. Although no opinion is advanced as to which is considered the preferred procedure, the data presented indicate that immersion at 140 F. is by far the most severe. The excessive stripping results obtained with the cutback asphalts at this temperature of immersion suggest that this immersion temperature should be applied only to asphalt cements and that a more moderate temperature is better suited for the cutback asphalts. An immersion temperature of 100 F. following curing at 77 F. appears a little unusual, wheres the reverse would appear more logical.

The paper favors the axial-compression stability test over the static immersion or modified Oberbach test because from the static immersion test there may result "a favorable report on an antistripping admixture when, as a matter of fact, this agent might do more harm than good in a bituminous concrete mix." This opinion is somewhat at variance with that expressed in another part of the paper discussing the measurement of stripping resistance by the compression test in which it was concluded "this is in general accord with the modified Oberbach stripping result for these same materials—generally speaking, this accord obtains throughout with most of the combinations." However, no specific tests performed with the same aggregate treated in the same way for comparing the static immersion and axial-compression tests are recorded in this paper so that the preference of the author for the compression test must be based on other considerations than those contained in the given experimental data. Until such information is made available, it may be concluded that the results of the static immersion test are at least equal in value to those from the axial-compression.

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[1] Esso Laboratories, Process Div., Standard Oil Development Co., Elizabeth, N. J.

In common with most work on asphalt additions, the laboratory results have not yet been correlated with field experience. This situation has been amply described by the author.

MR. WALTER C. RICKETTS[2] (*by letter*).—While the prime objective of the overall investigation was in connection with war, the laboratory study of antistripping admixtures discloses that the work accomplished is applicable to asphalt pavements constructed in time of peace.

It is believed that a careful digest of the paper merits the following deductions:

(*a*) There is a need for a standard laboratory method which has been properly correlated among cooperating laboratories for reproducibility to evaluate the efficiency of an antistripping agent under varying conditions.

(*b*) Terms such as "wetting power" used by the author and others applicable to antistripping agents require standardized definitions. For instance, the usual meaning of the terms "hydrophilic" and "hydrophobic" when applied to wet aggregates as compared with dry aggregates may be misleading.

(*c*) Any investigator who is contemplating a laboratory study of a given additive should base his evaluation of the additive's efficiency by comparing the asphaltic mixture to be used in the construction with the same mixture in which the additive has been properly introduced. This will permit the aggregate as a whole to be considered, as well as any detrimental effects that the additive may have on the physical characteristics of the asphaltic material.

(*d*) The laboratory study has provided much valuable information which may serve as a guide for subsequent investigations.

MR. L. C. KRCHMA[3] (*by letter*).—Mr. Sprague's paper is a most welcome addition to our knowledge on stripping and water resistance. We heartily agree that the conventional stripping tests are inadequate and accordingly we were interested in comparing his results in this report with similar ones reported by Krchma and Loomis.[4] Over and above this, Mr. Sprague's paper affords us an opportunity to examine the validity of the W_s constant developed in the paper mentioned above. It permits us to examine additional phases of quantitative water-resistance determinations and, finally, it allows further study of the antistripping agent problem and the often unrecognized complications that exist.

The W_s constant referred to above was based on the work reported in 1943 employing testing techniques essentially those used by Mr. Sprague here and by Messrs. Pauls and Rex.[5] At that time the relation of the water-susceptibility constant referred to as W_s was developed. This constant was the rate at which a bituminous surface lost bearing capacity, expressed as the reciprocal of the compressive strength in pounds per square inch. As the reciprocal of the compressive strength plotted against immersion time was a straight line, this constant represented the slope of this line and could be expressed by Eq. 2 of our 1943 report,[4] or as suggested by Mr. Holmes in his discussion of the paper.

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[2] Corps of Engineers, U. S. War Dept., Washington, D. C.

[3] White Eagle Div., Socony-Vacuum Oil Co., Inc., Kansas City, Mo

[4] L. C. Krchma and R. J. Loomis, "Bituminous-Aggregate Water Resistance Studies," *Proceedings*, Assn. Asphalt Paving Technologists (1943).

$$\frac{1}{C} = W_s t + \frac{1}{C_0}$$

where W_s is the water-susceptibility constant, C_0 the initial compressive strength in pounds per square inch, and C the compressive strength after t days of water immersion. The advantage of this approach is that the time of water immersion is taken into consideration. W_s also seems to lend itself better to the necessary analysis of the many variables. This work was done because it was felt a more direct method of determining water resistance was necessary, in keeping with the better design of highway surfaces now required.

Since Mr. Sprague reports stabilities of identical systems after saturating and 24 and 48-hr. immersions, it is interesting to note that in several instances the constants calculated for both conditions were of the same order of magnitude as would be expected if the W_s constant were valid. Table X, values of RC-2 bound dry pit run gravel, gives calculated values of the W_s constant for the 24 and 48-hr. immersion times of 0.012 and 0.011, respectively. On the other hand, RC-2 bound quartzite, Table XIII values of 0.0014 and 0.011 for 24 and 48 hr., show poor agreement. But an examination of the data shows the dry blank stabilities for the 24 and 48 hr. as 1131 and 750, respectively. Such differences in the initial strengths place the weaker specimens at a disadvantage and result in higher susceptibility.

The MC data also tend to show deviations. This is quite likely due to the lower strengths of the specimen. It was felt at the time the W_s constant was originally proposed that the limitations of the simple compressive strength test required emphasis on compressive strengths that were higher than 50 psi. (This would correspond to 350-lb. stabilities as reported by Mr. Sprague.) It seems we were too conservative, as recent data indicate that compressive strengths below 50 psi. can occasionally be used. Still, very weak systems can be expected to give too low a compressive strength, as many of them are so weak as to tend to fall apart under their own load. Hence, the forces applied by the operator and reported are quite likely only a fraction of the forces causing failure. Under such circumstances the compressive strengths would be low and unreliable and would give too high a W_s. A good example of this is the MC-2 cutback data of Table IX, where the constant for 24-hr. immersion is 0.16 and the 48-hr. value is 0.027. This poor agreement would be expected on the basis of Mr. Sprague's observations, namely, that the 24-hr. specimen started to crack under its own weight. (Incidentally, the initial strength of the original blank for these specimens is only 267 lb. as compared to 474 lb. for the specimen used in the 48-hr. test, further adding to the poor agreement.) In general, Mr. Sprague's results tend to reaffirm our belief that the simple compressive strength test technique employed by ourselves and also Mr. Sprague and others is probably adequate except for the weakest systems, and that the results can most usefully be expressed in terms of the W_s constant or similar. For weak systems, more ideal testing techniques seem necessary. Such tests would require, quoting from our original paper, "the use of a technique by which the effect of water upon the various components contributing to the stability of mix could be independently evaluated." A stabilometer might be a solution.

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[5] J. T. Pauls and H. M. Rex, "A Test for Determining the Effect of Water on Bituminous Mixtures," Bur. Public Roads, Vol. 24, p. 115 (1945).

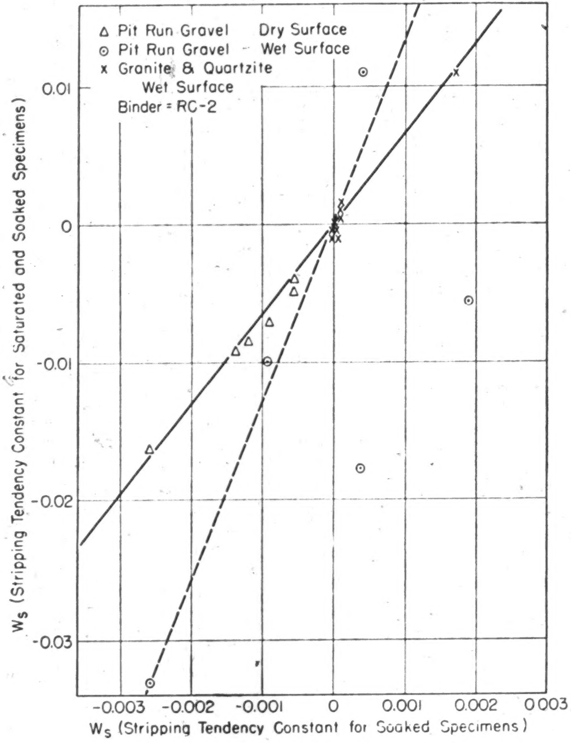

FIG. 10.—Comparison of W_s for Saturated and Immersed *versus* Immersed Specimens. (Based on Sprague's paper.)

Another feature of Mr. Sprague's work that is of interest regarding the testing technique is the acceleration imparted to the system by saturating and immersing as compared to straight immersion. Figure 10 represents a plot of the W_s constants for identical specimens saturated and soaked *versus* merely soaked. The W_s constant was used because it permitted a direct comparison of systems not tested for the same immersion times; that is, 24 or 48 hr. on one hand and 26 or 60 days on the other. The data shown include all the results, irrespective of the agents and quantities, but do not indicate the aggregate and the aggregate condition. Still, there are many types of systems and only rough generalizations seem permitted. The W_s values for saturated and soaked specimens for dry surface pit run gravel are some six to seven times larger than those obtained by simple immersion. The wet-surfaced pit run gravel saturated and soaked W_s values are roughly ten to twelve times the straight immersion values. The quartzite and granite data cover a very narrow range and, with the exception of one value, indicate the saturated and soaked W_s values are probably ten times larger. It would seem, then, that the accelerated test technique employed does not always give results that are uniformly comparable with the straight immersion results without some sort of a factor. This difference points up the problem of maintaining a normal balance among the important factors under accelerated conditions. Further correlation with field failure may be necessary to indicate which of the two techniques is most reliable.

It is interesting to note in Fig. 10 that the W_s ratio line for saturated and soaked *versus* simple immersion passes through zero with fair agreement. This is reasonable, since W_s is a rate of change of the reciprocal of the strength on immersion and it would be expected that the zero rate of change would still be zero even under accelerated conditions. It is also of interest to note the increase in the strength that occurs on immersing some bituminous aggregate specimens. Originally, when first noted, this increase caused us some concern. That these negative values for W_s increase under accelerated test conditions rules out the possibility that these results are accidental and it is quite evident that certain systems do increase in strength in the presence of water. The mechanism whereby this occurs offers interesting opportunities for speculation.

We note Mr. Sprague prepared specimens by straight compression, 2000 psi. using a floating mold. In our work we had found marked differences in the way specimens swell, depending upon whether compacted by straight compression, 2000 psi., or by vibration. The explanation given was aggregate failure under compression. We have not determined W_s on specimens prepared by straight compression but on the basis of the swell results we are of the opinion W_s will be higher and is a feature warranting some consideration, particularly if aggregate failure under compression is greater than under actual service.

We should be interested in knowing whether the water in which Mr. Sprague immersed the specimens was held at 77 F. We should be further interested in knowing whether Mr. Sprague had occasion to examine the effect of this water temperature on the stabilities after immersion. It would seem reasonable to assume that higher water temperatures during immersion would hasten the loss of stability and might offer a means of accelerating the failure. Of course if the water temperature has an effect on the rate the specimen breaks down, careful temperature control is necessary. We have assumed that this is the case, but other than doing work at 77 F. have not gone any further. We should also be interested in Mr. Sprague's observations on the effect of alternate wetting and drying cycles, noting whether the specimen once wetted returns to its original dry strength on drying or whether it progressively deteriorates. Such an approach might closely simulate field conditions. Again, we have not had occasion to examine this, but believe that it offers interesting possibilites for investigation.

The beneficial effect of age of the asphalt film on water resistance was commented on by Mr. Sprague. This feature was examined for one aggregate in our 1943 report.[4] It showed that the improvement in the W_s constant—that is, in water resistance—was quite rapid the first few days, but that as the specimen aged, the rate of improvement progressively became less and tended to reach a limiting value beyond 100 days.

This represents limited information but, such as it is, indicates that at least a year under ideal conditions would be required for a poor aggregate to reach an acceptable water-resistance stage. It will be of interest to establish what the improvement with age situation may be when antistripping agents are used.

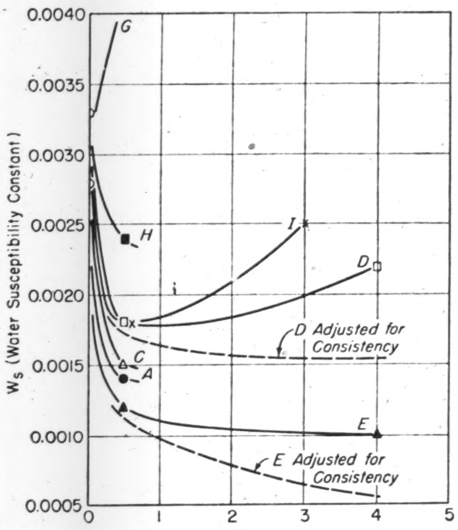

FIG. 11.—Effect of Antistripping Agent Quantities on W_s. (From Sprague's Table XII.)

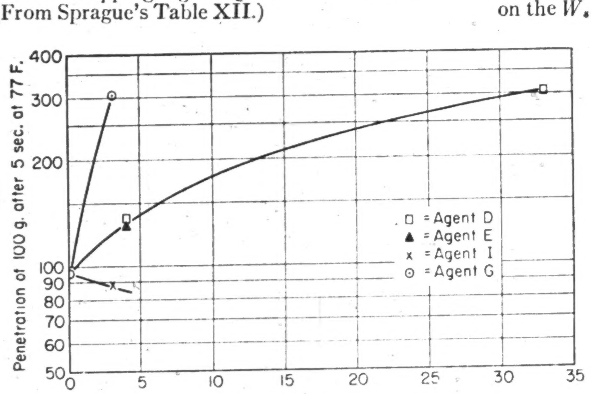

FIG. 12.—Effect of Antistripping Agent Additives on the Penetration of AC-8. (From Sprague's Table III.)

The effect of antistripping agent quantities is, of course, of considerable interest. Mr. Sprague's work, I believe, shows some of the anomalies that certainly exist and bears out some of the limited work that we have had occasion to do. We have found instances where stripping agents were not beneficial irrespective of the quantity of agent employed, and some instances where the agents actually made aggregates worse.

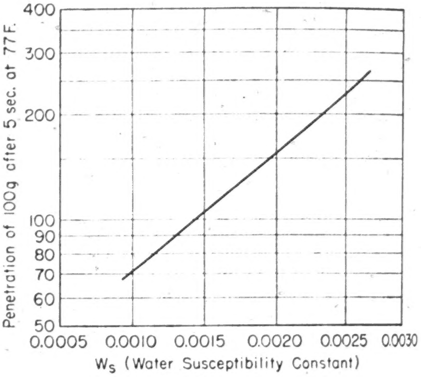

FIG. 13.—Effect of Binder Consistency on the W_s.

There did not appear to be any simple rules by which these agents could be used and it seemed that a better understanding of the situation was necessary before the most economic and optimum results would be obtained. The data in Table

XII of Mr. Sprague's report lent themselves to examination of the effect of agent quantities, as the blank and the modified binders were tested under identical conditions, except for immersion times which the use of W_s takes into consideration. This permitted the preparation of Fig. 11 of this discussion. It is evident that for agents D and I distinct optimum quantities are required. Agent E indicates little or no improvement beyond the initial ½ per cent.

One likely reason for the observed optimum is the effect the agent has on the consistency of the binder. The dotted lines of Fig. 11 represent the data adjusted for the change in the consistency of the binder. This was done on the basis of the reported changes in penetration per Table III of Mr. Sprague's paper (here summarized in Fig. 12) and the relationship of penetration to water susceptibility (Fig. 13, based on the data covered by Krchma and Loomis in their 1943 report[4]). There may be some question of the accuracy of this adjustment, but lacking better means, it still seems that even after correcting for consistency the greatest improvement occurs in the initial small percentages of agent added. In the case of agent I the adjustment was too small to be shown and the original line as drawn still applies. From this it seems evident that the quantity of antistripping agent must be carefully chosen for the aggregate as well as the binder plus probably some adjustments for the gradation.

Another feature that is of interest is the effect of the agent on the consistency of the binder, as this has considerable bearing on the stability of a surface as well as stripping. Comparing the results for the RC-2 residues, per Mr. Sprague's Table IV, with those for the AC-8 summarized in Fig. 12 based on Table III, it appears that agents D and E soften the RC base more rapidly than the AC-8. On the other hand, agent G greatly softened AC-8 but seemed to harden the RC-2 distillation. Again, this tends to point up the difficulties in working on problems of this nature and the care that must be taken in interpreting the results. Certainly Mr. Sprague is to be complimented for his careful evaluation of the changes that occur in the binder due to adding agents.

The addition of the agents and the fact that they can result in lower stabilities raises the problem of whether they are always entirely beneficial, irrespective of the possible improvement in the stripping tendency per cent as employed by Mr. Sprague. I believe he touched on this matter; namely, the reduction in the stability of the system due to the use of the antistripping agents in part nullifies the improvement in stripping resistance due to the agent. Certainly it would seem that as the original strength of a surface is decreased for any reason whatsoever higher water-resistance requirements are necessary. Further, it is a question of whether a system with a stability of say, 1400 lb. and a 50 per cent stripping tendency for 24 hr. is a poorer system than the same system treated with an antistripping agent to the extent that its initial dry strength drops to 700 lb. but with a stripping tendency per cent of zero. Both systems after water immersion have the same stability. In this instance, it would appear that the agent was not an unmixed blessing if the surface normally requires stabilities higher than 700 lb.

Carrying the above example further, were there two systems, one having a 1400-lb. stability on the basis employed by Mr. Sprague (200 psi. compressive strength for a 3-in. diameter specimen) and the other a 700-lb. stability (100 psi. strength) and both had a stripping tendency of 50 per cent, both would be rated equal in their water resistance, even though their respective stabilities after immersion were 700 and 350 lb. However, the lower stability surfaces should have higher water-resistance requirements, as they are closer to whatever minimum stability is required for a surface. Accordingly, in the above example the weaker of the two would give a surface more subject to water failure. In this respect the W_s concept gives a somewhat different picture, as the above systems would have constants of 0.005 and 0.01, respectively. This rating, although with no direct precise correlation with the field, certainly seems more reasonable. However, it is interesting to note that were the weaker of the two systems corrected through the use of agents to give it 0.005 W_s value, after 24 hr. it would have a strength of only 665 psi. On this basis it would appear that a second consideration of water resistance is still necessary; namely, have systems with low W_s plus high initial strengths. As some of the addition agents soften the binder and reduce the initial stabilities, agents in all instances will not necessarily permit this, and it accordingly seems desirable from a design point of view to choose aggregates and binders and construction conditions wherever possible to give high initial strengths irrespective of whether an agent is employed. Of course in this respect it will be of interest to establish whether the weakening effects of some of the agents are temporary or permanent, whether the improved water resistance is permanent, and whether the resistance improves with age as with an ordinary mix.

We hope information on these variables will ultimately be available.

MR. JOHN C. SPRAGUE (*author's closure*).—The comments and test data presented by Messrs. Holmes, Ricketts, and Krchma form a welcome addition to the author's paper. With regard to Mr. Holmes' observation relative to the incorporation of the additive at the refinery, the author can only comment that it might be risky unless the aggregate were predetermined and its characteristics, as well as the character and function of the mix, fully investigated beforehand. To his knowledge there is no antistripping agent on the market that has an over-all efficiency from standpoint of wetting power, stripping resistance, and stability retention for all aggregates under all conditions. Presupposing proper precautions, however, it might be more advantageous to add the agent at the refinery—certainly it would preclude the possibility of changing drastically the original characteristics of a bitumen, by so modifying the bitumen itself so that it would meet pertinent requirements after the agent had been added. For example an AC-6 asphalt might well have the characteristics of an AC-8 asphalt after the optimum amount of a given agent had been added. Also, everything else being equal, adding at the refinery would be more economical. It is considered that curing at 77 F., followed by immersion at 100 F., was the most realistic condition of test for the static immersion.

With regard to Mr. Holmes' comments on static immersion *versus* axial-compression test it can only be reiterated that the latter brought out weaknesses in the mixtures which the former failed to reveal. In general both tests did show the same trends in so far as stripping tendency is concerned, although there were a few exceptions. But the axial-compression test revealed shortcomings from standpoint of adhesion (as opposed to coverage) and stability which static immersion did not. The author is in complete agreement with

Messrs. Holmes and Ricketts that the terms hydrophilic and hydrophobic should be given a clearer meaning, and probably a more limited one; further, they should be supplemented with additional qualifying terms. And as Mr. Ricketts stated, other terms need to be standardized. Mr. Ricketts' deductions are valued in that they so cogently sum up and express the spirit and substance of this investigation. Standardization of method definitely is a first "must." Certainly if a proposed additive produces mixtures with substantially lower stability than the plain bituminous mix itself, it has questionable value; on the other hand, if it exhibits superior antistripping performance on mixtures subjected to severe soaking conditions, if it will not lower stability below a minimum structural optimum, then some sacrifice to stability might well be afforded.

Mr. Krchma's comparisons were very much to the point—only by such comparative data can a suitable standardized procedure be eventually developed which will adequately cope with all facets of this many-sided problem. With regard to his inquiry, the immersion water was held at 77 ± 5 F. Mention of this was inadvertently omitted in the procedure, but was touched upon in the appendix. No measurable effect was noticed in this narrow range in temperature of the bath water. There is no question but that appreciably higher bath temperatures would accelerate failure of the specimens—this is indirectly evident in the static immersion tests. While no actual determinations were made in this investigation to evaluate losses due to alternate wetting and drying, as compared with constant immersion over a long period of time, it was assumed that the effect of summation of wetting periods in the former would about equal the latter. It is not clear how the mix could appreciably recover during the "drying" period unless it were heated. It might hold up better until the bond was broken, after which failure might actually be more rapid than it would be under constant immersion conditions. This is conjecture, of course, and needs to be evaluated by actual tests.

DEVELOPMENT OF A METHOD OF TEST FOR SOIL BITUMINOUS MIXTURES*

By E. O. Rhodes[1] and P. F. Phelan[1]

Synopsis

During the past five years, a group of technical representatives of leading producers of cutback asphalt, emulsified asphalt, and tar has collaborated in the development of a method of test for soil bituminous mixtures. This was done to overcome the confusion caused by the present multiplicity of tests for bituminous stabilized soils.

In brief, the proposed test method consists of submitting 2-in. diameter by 2-in. high specimens to a 7-day absorption test in 1 in. of water, followed by a test in which a 1⅛-in. diameter plunger travelling at the rate of 1 in. per minute extrudes the mixture through a 1⅛-in. diameter orifice. Particular care has been exercised to provide control features—in the preparation of mixtures, in the forming and handling of specimens, and in the testing for absorption, expansion, and strength characteristics—that eliminate many of the factors responsible for poor reproducibility of test results in some other methods.

The proposed test method is now under consideration by a joint subcommittee of A.S.T.M. Committees D-4 on Road and Paving Materials and D-18 on Soils for Engineering Purposes. This paper is a report by the Chairman and Secretary of the Bituminous Producers' Cooperative Research Committee on the work performed in the development of the test method.

In March, 1941, technical representatives from six bituminous producer organizations met with representatives of the Civil Aeronautics Authority to discuss the various types of test that might be used for testing soils after treatment with bituminous materials. At that time it was realized that progress in the field of bituminous soil stabilization would be hampered if each bituminous producer's laboratory and each state testing laboratory continued on its own way, developing its own individual method of test. The C.A.A. suggested that the producers consider the situation with the thought in mind of suggesting a mutually acceptable procedure that might be followed by the C.A.A. in its testing of bituminous stabilized soils. Since that time, this group of bituminous producers, calling itself the Bituminous Producers' Cooperative Research Committee, has worked together to develop a simple, direct method of test which can be used for testing soil-bituminous mixtures containing emulsified asphalt, cutback or other liquid asphalt, or tar.

The personnel of this committee included Messrs. C. L. McKesson and P. E. McCoy of the American Bitumuls Co.; B. A. Anderton and J. W. Donegan of the Barrett Division, Allied Chemical and Dye Corp.; E. W. Klinger, Standard Oil Co. of New Jersey; J. C. Roediger and A. Holmes of the Esso Laboratories, Process Division; Prevost Hubbard of

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] Manager of Development and Highway Engineer, respectively, Koppers Co., Inc., Tar Products Div., Pittsburgh, Pa.

the Asphalt Institute; E. O. Rhodes, A. C. Havens, and P. F. Phelan of Koppers Co., Inc. To each cooperating laboratory in the group, the investigation of the various test procedures and the research work performed in the development of this method is the equivalent of one man's full time for 20 months, including the fabrication and testing of over 2000 specimens. During this 5-yr. period, this group has held twelve meetings, which represent over 75 hours of joint discussion of test results, apparatus design, refinements of test procedure, and other pertinent problems.

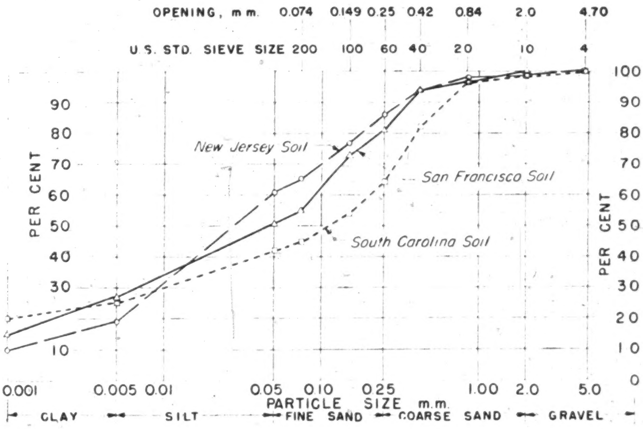

Fig. 1.—Grading of Soils Used in B.P.C.R. Tests.

TABLE I.—COMPARISON OF B.P.C.R. TEST WITH COLLABORATORS' OWN TEST METHODS.

Laboratory	Maximum Particle Size	Size of Specimen		Compaction Loading			Strength Test Apparatus			
		Inside Diameter of Forming Mold, in.	Compacted Height of Specimen, in.	Total Load, lb.	Time Held, min.	Unit Load, psi.	Inside Diameter of Test Mold, in.	Diameter of Test Plunger, in.	Inside Diameter of Extrusion Orifice, in.	Speed of Plunger Movement During Test, in. per min.
Bitumuls	¼ in.	2	2	3 000	1	954	2.06	2.03	1.125	0.4 to 0.5
Barrett	No. 10	2.00	1.00 +0.05	2 000	2	636	2[a]	1.125	1.125	1
Esso	No. 4	2	0.9 to 1.0	10 000	2	3183	2.02	2	1.75	2.4
Koppers	No. 10	1	approx. 0.25[b]	300	0.5	382	1	0.25	0.4	3
B.P.C.R.	No. 4	2.000 ±0.001	2.00 ±0.05	6 000	2	1910	2.020 ±0.001	1.125 ±0.001	1.125 ±0.001	1

[a] Mold split into three sections, tightened to fit specimen prior to testing.
[b] Specimen contains 8.0 g. dry soil.

At the first meeting of the committee, a review of the methods of test then in use in each of the collaborating laboratories showed that all laboratories were using an extrusion type of test (Table I). It was noted that American Bitumuls Co. was using a 2-in. diameter by 2-in. high specimen; the Barrett Div., Esso, and The Asphalt Inst. were using 2 by 1-in. specimens; and Koppers was using a 1 by $\frac{1}{4}$-in. specimen. The compaction loading varied from a total load of 300 to 10,000 lb. The test plungers were either larger than, of the same diameter as, or smaller than the extrusion orifice.

At the conclusion of its first meeting, the committee had agreed that:

(*a*) Rigid and nonrigid base mixtures cannot be evaluated by the same test procedures because the two types are fundamentally different in design and performance,

(*b*) When nonrigid base mixtures are subjected to any type of load-bearing or strength test, the test specimens should have some type of side support if reproducible results or indications of service behaviors are to be obtained, and

TABLE II.—ANALYSIS OF SOILS USED IN B.P.C.R. TESTS.

	South Carolina Soil	San Francisco Soil	New Jersey Soil
Screen Analysis (Wet)			
Passing No. 4 sieve, per cent	100	100	100
No. 10 sieve, per cent	99	100	99
No. 20 sieve, per cent	97	97	98
No. 40 sieve, per cent	82	94	94
No. 60 sieve, per cent	64	81	86
No. 100 sieve, per cent	54	73	77
No. 200 sieve, per cent	45	55	65
Gravel (over 2.00 + mm.), per cent	1	0	1
Coarse Sand (2.00 to 0.25 mm.), per cent	35	19	13
Fine Sand (0.25 to 0.05 mm.), per cent	22	30	25
Silt (0.05 to 0.005 mm.), per cent	17	24	42
Clay (below 0.005 mm.), per cent	25	27	19
Colloids (below 0.001 mm.), per cent	20	15	10
Textural Description	Sandy clay	Sandy clay loam	Sandy silt
Liquid Limit	29	29	31
Plastic Limit	17	17	20
Plasticity Index	12	12	11
Field Moisture Equivalent	..	20	25
Shrinkage Limit	14	14	15
Shrinkage Ratio	..	1.9	1.8
Soil Classification—PRA	A-2-4	A-2-6	A-4-6
Group Index	3	5	6
Proctor Optimum Moisture, per cent	12	15	16
Proctor Dry Soil Density—lb. per cu. ft.	122.5	115.0	112.7

(*c*) Bituminous stabilized soil mixtures preferably should be evaluated by some type of extrusion test wherein failure occurs due to excessive plastic flow of the mixture under test.

Since that time, a large volume of work has been performed, as indicated in the brief résumé which follows:—Three soils were selected for use in cooperative tests—a South Carolina sand clay, a San Francisco sandy clay loam, and a New Jersey sandy silt (Table II). The grading curves of the three soils are plotted in Fig. 1. Each laboratory was supplied with over one quarter of a ton of each of the three soils, and large supplies of stabilizer emulsion, cutback asphalt, and tar. Mixtures for cooperative testing were selected after each collaborator tested each of the soils for optimum percentage of moisture and bituminous stabilizer, using his own preferred method of test.

When the committee's method of test was originally outlined, it provided for testing 2-in. diameter by 2-in. high and 2 by 1-in. specimens, both uncured and cured at 140 F., after being subjected to capillary absorption or $\frac{1}{2}$-in. immersion for 7 days. All specimens were tested for strength by extrusion through a $1\frac{1}{8}$-in. diameter orifice by means of a $1\frac{1}{8}$-in. diameter plunger moving at a constant rate of 1 in. per minute. Other types of absorption tests—complete immersion and almost complete (flush with the surface) immersion—and the use of additional plunger-orifice combinations were also investigated by some of the collaborating laboratories. Density, shrinkage and loss on curing, swell during absorption, and other characteristics of the test specimens were measured. In this series of tests, each laboratory was required to mold and test approximately 500 specimens. The results of these tests revealed that each laboratory could duplicate its own results quite well, and that fairly good agreement among laboratories was obtained on specimen densities, molding moisture contents, and amounts of absorbed moisture. However, discouragingly poor agreement for the strength results among the different laboratories was obtained.

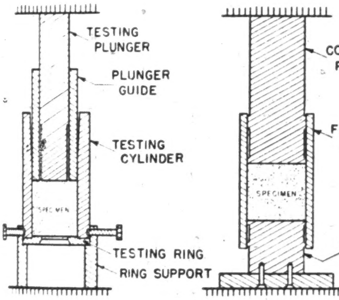
TESTING PLUNGER
PLUNGER GUIDE
TESTING CYLINDER
SPECIMEN
TESTING RING
RING SUPPORT
COMPACTION PLUNGER
FORMING MOLD
SPECIMEN
COMPACTION PLUNGER

FIG. 2.—B.P.C.R. Testing Assembly (Left) and Molding Assembly (Right).

After much thought and extended discussion concerning the probable reasons for the wide differences in strength results, it was decided that different laboratories should prepare the *same mixture* and send specimens of the molded mixture to the other laboratories for testing. It was intended that this should point to any differences in mixing, molding, or curing procedures; and to differences in absorption or strength-testing equipment. Each of three laboratories formed 40 specimens (2-in. diameter by 2in. high) from a mixture of the New Jersey soil with 6 per cent water and 9 per cent MC-2 cutback asphalt. After curing, the specimens were distributed—eight specimens from each batch of 40 going to each of the five cooperating laboratories. All mixtures were prepared on the same day, and the specimens were formed, cured, shipped, and tested in accordance with previously agreed-upon specific instructions covering procedures and work schedules. From each group of eight specimens, three were tested (on a specified day) for strength before absorption; and five were subjected to 7-day absorption in ½ in. of water before being tested for strength. Very complete data were recorded, including room temperature at time of molding specimens, absorption-cabinet temperature during the absorption period, room temperature at the time the strength tests were performed, and depth of penetration of the plunger at point of maximum strength of the specimen.

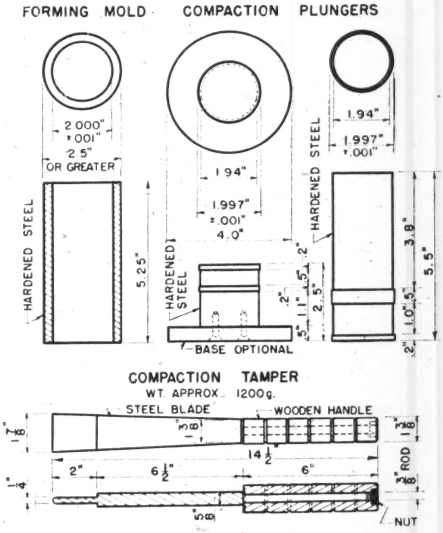
FORMING MOLD
COMPACTION PLUNGERS
2.000" ±.001"
2.5" OR GREATER
HARDENED STEEL
5.25"
1.94"
1.997" ±.001"
4.0"
HARDENED STEEL
.2"
.5"
.2"
1.1"
.5"
2.5"
BASE OPTIONAL
1.94"
1.997" ±.001"
HARDENED STEEL
3.8"
5.5"
.5"
1.0"
.2"
COMPACTION TAMPER
WT. APPROX. 1200g.
STEEL BLADE
WOODEN HANDLE
1 7/8"
1 3/8"
1 3/8"
14 1/2"
2"
6 1/2"
6"
3/8" ROD
1 1/4"
5/8"
NUT

FIG. 3.—B.P.C.R. Molding Equipment.

As a result of these tests, a number of refinements were made in the test procedure. To insure comparable densities, a close tolerance was placed on the moisture content at time of molding; a pre-compaction technique using a blade type tamper was agreed upon; a compaction loading rate was specified; and a tolerance was placed on the compacted height of specimen. It was also agreed that specimens should be immersed to one half their depth during the absorption test. The data indicated that three specimens, instead of five, were sufficient to obtain a good average result for testing purposes.

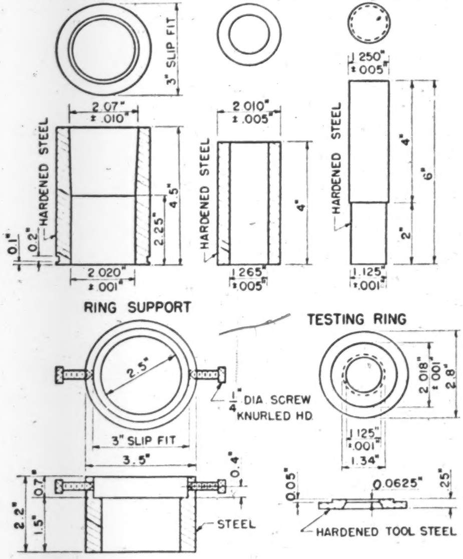

Fig. 4.—B.P.C.R. Testing Equipment.

With the test method thus revised, another series of tests was performed. Each laboratory formed eight 2 by 2-in. specimens of a specified mixture, cured them to constant weight at 140 F., tested three for strength before absorption, and tested the remaining five for strength after 7 days' absorption in 1 in. of water. The test results from the various laboratories checked very well as far as densities, absorption, and change in volume during absorption were concerned, but again the final strength values were at greater variance than desired.

Based on the above results, close tolerance limits were placed on the dimensions of the forming and testing equipment, and the entire test method was again critically reviewed. The oven-curing procedure for emulsified asphalt-treated specimens was changed from drying to constant weight, to drying until 80 to 90 per cent of the molding moisture had been removed. This was intended to eliminate the possibility of excessive differences in residual moisture contents when drying to constant weight in different laboratories.

The test procedure, as then revised, was submitted to the Joint D-4, D-18 Subcommittee on Tests and Specifications for Stabilized Soils, at the June, 1944, meeting of A.S.T.M. It was also submitted to the committee for the Symposium on Soil Testing Methods, and was included in the compilation "Procedures for Testing Soils," published in September, 1944. Apparatus setups and dimension details of the molding and testing equipment are shown in Figs. 2, 3, and 4.

Since close tolerance limits had been placed on the dimensions of the forming and testing equipment, and as none of the cooperating laboratories was then equipped with apparatus meeting these narrow limits, it was agreed that each collaborator would obtain such equipment and perform another series of tests. In this series, each of the three soils used in the former tests was mixed with a different bituminous stabilizer and tested in accordance with the latest revised procedure. Each laboratory prepared its own mixtures and formed its own specimens for testing. Completion of this series of tests was delayed on numerous accounts, chief of which were the shortage of laboratory personnel and the difficulty of getting accurate machine work done in shops overburdened with war projects carrying high priority ratings.

This final test series has been completed for some time. It is being reported now so that data will be available to the interested committees of A.S.T.M and other organizations that have this test method under consideration. The data and reports of this cooperative work are too voluminous to present in this paper. However, in the review of certain phases of the test method which follows, typical data are presented to show reproducibility, reasons for certain revisions or refinements in test procedure and equipment design, and some of the difficulties encountered in the development of the test method.

REVIEW OF DETAILS IN DEVELOPMENT OF THE TEST METHOD

Apparatus:

Mixers.—Three different types of laboratory mixers were used in these investigations, and all were found satisfactory. Bitumuls used a Readco, Model P-12; Barrett used a Hobart Model C Mixer; while Esso and Koppers employed the Kitchen Aid Mixer, Model G.

Compaction.—It was found that compaction of some soils is benefited by preliminary tamping of the mixture in the mold before applying the compaction load. This pre-compaction tamping apparently serves to orient the particles so that greater uniformity of densification is obtained under the compaction load. The tamper used is that widely used in the Hubbard-Field test procedure; it is often referred to as the Hubbard-Field No. 1 Tamper (Fig. 3).

Differences in density among laboratories continued even with this pre-compaction tamping, until a definite procedure was specified, namely, that the tamper be dropped 25 times (upon the total material to be molded) from a height of 6 in. above the bottom of the specimen. To prevent damage to the top of the mold by the tamper, a collar of some type is placed on the top of the forming mold. The tamper is rotated between applications of the 25 tamps. Comparable densities are further assured by specifying that the compaction load be applied at a rate of 1-in. per minute movement of the compaction plunger—the same application rate used when testing for strength.

Strength Test Apparatus.—The design and close tolerance limits placed on the dimensions of the strength-testing apparatus insure that the specimen under test is centered over the testing orifice, which is centered in the testing mold, while the test plunger is centered (by means of a guide) directly over the specimen (Figs. 2 and 4).

In this cooperative work, both hydraulic and mechanical (screw-actuated) testing machines were employed. Bitumuls used a 90,000-lb. Southwark-Emery hydraulic machine and a 300,000-lb. Riehle screw-type machine. Barrett, Esso, and The Asphalt Inst. used a standard Hubbard-Field 10,000-lb. screw-type testing machine. The Asphalt Inst. also used a 100,000-lb. Baldwin-Southwark hydraulic machine. Koppers used a 20,000-lb. modified Carver hydraulic laboratory press and a 100,000-lb. Riehle screw-type testing machine.

Preparation of Soil:

The maximum aggregate size (passing No. 4 sieve) for use in the tests was chosen to conform to widespread current practice. It permits the use of field samples of reasonable size, yet allows test results within the range of testing equipment readily available in most bituminous laboratories. The soil is air-dried, rather than oven-dried, to prevent any change in soil characteristics that might be altered by drying at high temperatures.

Moisture Content of Soil.—The moisture content of the air-dried soil is determined by extraction with xylol in accordance with A.S.T.M. Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (D 95 - 40).[2] It was originally intended to determine the hygroscopic moisture content of the air-dried soil by oven-drying to constant weight. However, since the D 95 method is required in the determination of moisture contents of soil-water-bituminous mixtures, it was decided to employ also the D 95 method for the determination of moisture content of the air-dried soil—so that moisture contents would be on a comparable basis.

Preparation of Tar or Liquid Asphalt-Soil Mixtures:

In the preparation of mixtures containing liquid asphalt or tar, the desired amount of water (usually 0.25 to 0.5 per cent greater than that specified for molding) is added to the air-dried soil and mixed for 5 min. in the laboratory mixer, after which the mixture is aged in a closed container for 16 to 24 hr. A 2 or 3-min. mixing period is usually sufficient to distribute the moisture throughout the soil. This varies with different soil types, however, so a 5-min. mixing period was adopted as a safe standard.

It was found desirable to age the soil-water mixture in order to obtain uniform distribution of low percentages of moisture. The soil-water mix is made one afternoon, and aged overnight. The next morning, the aged soil-water mixture is then placed in the bowl of the mechanical mixer; the required amount of bituminous stabilizer is added and then mixed for a 5-min. period. Additional mixing with a rubber-tipped pestle may be required to produce a uniform mix, after which the soil-water-bituminous mixture is placed in an air-tight container for a 4-hr. period. This second aging period allows for better distribution of the bituminous material throughout the mixture, prior to the forming of test specimens.

Preparation of Emulsified Asphalt-Soil Mixtures:

Since the emulsified asphalt mixtures contain excess water, no advantage is gained by aging the soil-water or soil-water-bituminous mixtures. Instead, the specified amount of mixing water is added to the air-dried soil in the mechanical mixer and mixed for 5 min. The specified percentage of emulsified asphalt is added, and mixing is continued for 5 to 10 min., until visual homogeneity is obtained. The mixture is then placed in tared, shallow pans and allowed to air dry (with frequent stirring to prevent crusting) to the specified molding moisture content. With some highly plastic soils, after the mixture has dried to approximately the plastic limit, it is remixed in the mixer, and is then placed again in the shallow pans to dry to the specified molding moisture content.

Molding Moisture:

Prior to molding specimens the soil-water-bituminous mixture is tested for water content by A.S.T.M. Method D 95, using xylol in the extraction. The moisture content must be within 0.15 per cent of the molding moisture specified. Thus, if the molding moisture is specified as 6.0 per cent, the mixture must be discarded if the determined moisture content is lower than 5.85 or higher than 6.15 per cent. This close tolerance is necessary since density, absorption, and strength values are so greatly affected by the moisture content at time of molding.

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[2] 1944 Book of A.S.T.M. Standards, Part II, p. 599; *Ibid.*, Part III, p. 293.

Forming and Measuring Specimens.—After initial compaction by 25 blows of the tamper falling freely from a height of 6 in. above the bottom of the specimen, final compaction is obtained by applying a 6000-lb. total load through a plunger travelling at the rate of 1 in. per minute. After holding the load for 2 min., the pressure is released and the specimen is removed from the mold to be weighed and measured for volume. The height of a specimen cannot vary by more than 0.05 in. from the specified height of 2.00 in. It was found that various laboratories could easily stay within a tolerance of ±0.02 in.

TABLE III.—DRY SOIL DENSITIES AT OPTIMUM MOISTURE OF PROCTOR VERSUS B.P.C.R. METHODS.

Soil	Proctor Method		B.P.C.R. Method	
	Optimum Moisture, per cent	Density, lb. per cu. ft.	Optimum Moisture, per cent	Density, lb. per cu. ft.
South Carolina	12	122.5	9	127.3
San Francisco	15	115.0	10	125.8
New Jersey	16	112.7	10.5	125.6

Density. — Throughout this test method all percentages of moisture, bituminous material, and absorption are based on the calculated weight of dry soil in the specimen. Thus, a mixture containing 6 per cent moisture and 9 per cent bituminous stabilizer is considered to contain 100 parts by weight of calculated dry soil, 6 parts by weight of moisture, and 9 parts by weight of stabilizer. Too, the percentage of absorption is calculated as the gain in weight during absorption divided by the calculated dry weight of soil in the specimen when molded, rather than by the weight of the specimen before absorption. On the same basis, the density of the specimen is always expressed as the calculated weight of dry soil in the specimen divided by the volume of the specimen. Thus, all moisture contents and densities are directly comparable, being based on the amount of dry soil in the specimen, regardless of its condition.

Table III shows the optimum moisture contents and dry soil densities of the three soils used in the cooperative tests, as determined by the standard Proctor and the B.P.C.R. compaction methods. The B.P.C.R. method results in appreciably lower optimum moisture contents and higher dry soil densities.

Curing Specimens.—Soil-asphalt and soil-tar specimens are usually tested in the uncured state. However, if the testing of cured specimens is desired, additional specimens may be cured to constant weight in a 140 ± 5 F. oven—constant weight being defined as the weight at which the loss during a 6-hr. period in the oven is 0.3 g. or less. The procedure provides for an initial curing

TABLE IV.—COMPARISON OF MOISTURE LOSSES DURING 140 F. OVEN CURING IN DIFFERENT LABORATORIES

San Francisco soil—8 per cent water—11 per cent emulsion (6.2 per cent asphalt), cured to lose 80 to 90 per cent of molding moisture.

Laboratory	Moisture When Molded, per cent[a]	Moisture Lost While Curing at 140 F., per cent	Loss of Molding Moisture While Curing, per cent	Residual Moisture Content, per cent	Absorption, per cent	Total Moisture Content After Absorption, per cent
Bitumuls	8.02	6.79	84.6	1.23	4.09	5.32
Koppers	7.87	6.42	81.5	1.45	3.72	5.17
Esso	8.12	6.41	79.0	1.71	4.40	6.11
Barrett	8.13	6.33	77.8	1.80	4.47	6.27

[a] Molding moisture determined by A.S.T.M. Method D 95–40, using xylol, 1944 Book of A.S.T.M. Standards, Part II, p. 599 and Part III, p. 293.

period of 40 to 42 hr. (a night, a day, and a night) followed by 6-hr. intervals in the oven until constant weight is attained. During this curing period some of the volatiles are lost from the tar or cutback asphalt, although all of the moisture is not lost.

For the soil-emulsified asphalt mixtures, a somewhat different procedure was found desirable. For these mixtures, the curing at 140 ± 5 F. is continued until 80 to 90 per cent of the moisture content at time of molding is lost. Table IV shows mixes made in four laboratories varying in molding moisture content from 7.87 to 8.13 per cent, a range of 0.26 per cent. After curing, during which 77.8 to 84.6 per cent of the molding moisture was lost, the residual moisture contents varied between 1.23 and 1.80 per cent, a range of 0.57 per cent. Thus, any differences in efficiencies of drying ovens would not appreciably affect the final test results, but would be reflected in the varying lengths of time required for curing in the different laboratories.

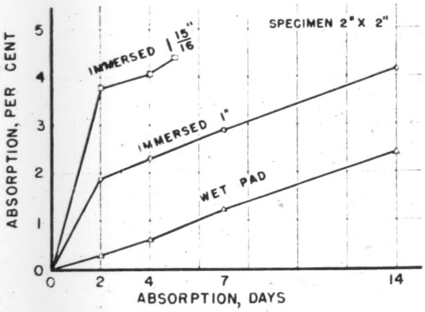

FIG. 5.—Comparison of Various Absorption Test Methods.

Water Absorption Test.—Four types of absorption test were tried in the course of this investigation: capillary absorption, immersion to half depth, immersion to full depth with the exception that the surface of the specimen was not covered, and complete immersion. In the capillary absorption tests, two laboratories used perforated metal plates with the water level adjusted to the top of the plates, while other laboratories used wet sand bases or porous stone blocks with the height of water adjusted just below the surface. Thus, in two cases the specimens stood in a thin film of water, while in the others they merely rested on a very damp surface. This

TABLE V.—EFFECT OF SLIGHT VARIATION IN INSIDE DIAMETER OF TESTING MOLD.

New Jersey soil—6 per cent water—9 per cent MC-2, cured at 140 F., then tested.

	Molded Dry Soil Density, lb. per cu. ft.	Diameter of Bottom of Specimen, in.	B.P.C.R. Strength, psi. Inside Diameter of Testing Mold	
			2.000 in.	2.021 in.
	115.1	1.99	8400	
	115.2	1.99	8100	
	115.4	1.99	8000	
	115.6	1.98	8900	
	115.5	1.99	9100	
Average	115.4	1.99	8500	
	115.4	1.98		6300
	115.3	1.99		6600
	115.3	1.99		6200
	115.3	1.99		6200
	115.2	1.99		6000
Average	115.3	1.99		6260

TABLE VI.—EFFECT OF SLIGHT VARIATION IN INSIDE DIAMETER OF TESTING MOLD.

New Jersey soil with MC-2, cured at 140 F., then tested.

	Molded Dry Soil Density, lb. per cu. ft.	B.P.C.R. Strength, psi. Koppers Laboratory, Inside Diameter, 2.000 in.	Bitumuls Laboratory, Inside Diameter, 2.019 in.	Esso Laboratory, Inside Diameter, 2.029 in.
	116.4	6400		
	116.3	6500		
	116.2	6300		
Average	116.3	6400		
	114.8		4410	
	114.9		4155	
	114.8		4600	
Average	114.8		4388	
	115.7			4450
	115.7			4100
	115.6			3850
Average	115.7			4100

affected the absorption results appreciably.

With complete immersion some difficulty was experienced due to the upper edges of the specimens spalling off—apparently due to the surrounding water preventing the escape of air from the inside of the specimen.

TABLE VII.—LENGTH OF TIME REQUIRED FOR B.P.C.R. TEST.

TAR AND CUTBACK ASPHALT TREATED SOILS

Step 1. Pulverize air-dried soil
2. Determine moisture content of air-dried soil
3. Prepare soil-water mixture
4. Age soil water mixture overnight (16–24 hr.)
5. Prepare soil-water-bituminous mixture
6. Age soil-water-bituminous mixture (4 hr.)
7. Determine moisture content of aged soil-water-bituminous mixture
8. Form specimens, weigh, and measure for volume
9. Place specimens in absorption cabinet for 7 days
10. Remove specimens from absorption cabinet, weigh, and measure for volume
11. Test specimens for B.P.C.R. strength value

Steps 1, 2, 3, 4	1 day
Steps 5, 6, 7, 8	1 day
Steps 9, 10, 11	7 days
	9 days—usual total time required

Optional—If Cured Specimens are Desired

Step 8.1 Place specimens in 140 F. oven for 40 to 42 hr.
8.2 Remove from oven, cool, and weigh
8.3 Place specimens in oven again for 6 hr.
8.4 Remove from oven, cool, and weigh
8.5 Repeat Steps 8.3 and 8.4 until specimens have reached constant weight
(Usually Step 8.5 is unnecessary)

Steps 8.1 through 8.4	2 days—usual additional time required for curing
Step 8.5	1 day for each repetition—usually not required.

EMULSIFIED ASPHALT TREATED SOILS

Step 1. Pulverize air-dried soil
2. Determine moisture content of air-dried soil
3. Prepare soil-water-bituminous mixture
4. Air-dry soil-water-bituminous mixture to specified molding moisture
5 Determine moisture content of mixture
6. Form specimens, weigh, and measure for volume
7. Cure in 140 F. oven to remove 80–90 per cent of molding moisture
8. Weigh specimens and measure for volume
9. Place specimens in absorption cabinet for 7 days
10. Remove specimens from absorption cabinet, weigh and measure for volume
11. Test specimens for B.P.C.R. strength

Steps 1, 2, 3,	1 day
Steps 4, 5, 6,	4 days
Steps 7, 8,	5 days (including week-end)
Steps 9, 10, 11	7 days
	17 days—usual total time required

With test specimens immersed in different depths of water, absorption increased with the depth of immersion—almost complete immersion (water just below the top surface) giving the highest results. However, since almost complete immersion seemed much more severe a condition than would probably be encountered in service, and because of the difficulties encountered with capillary absorption and complete immersion, it was decided to perform the absorption test by immersing the specimens to one half their depth. Figure 5 indicates the rates at which maximum moisture contents are attained in three types of absorption test—capillary, immersion to one-half depth, and almost complete immersion.

Expansion.—The expansion value as defined in the B.P.C.R. test method is based on the assumption that, given complete immersion for sufficient time, the sample will expand in all directions in proportion to the change in diameter of the bottom of the specimen during the 7-day absorption test. Thus, it is an arbitrarily selected value which is based on the difference in the cube of the diameter of the immersed end of the specimen before and after test.

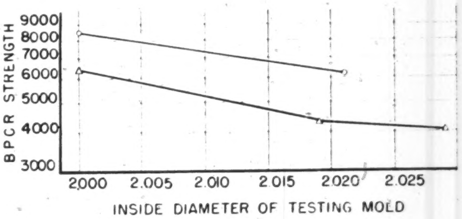

FIG. 6.—Effect of Slight Variation in Inside Diameter of Testing Mold.

Strength Test.—In performing the strength test, the specimen is placed in the testing mold which fits over the testing ring. Load is applied through the centered 1.125-in. diameter plunger moving at the rate of 1 in. per minute which extrudes the specimen through the 1.125-in. diameter orifice. Failure usually occurs after the plunger has moved a distance of $\frac{3}{8}$ to $\frac{5}{8}$ in. Tables V and VI show the effect of slight differences in the internal diameters of the test cylinders. For the data shown in Table V, all tests were performed in the same laboratory. Two sets of five specimens each, having the same molded density and the same diameter at the bottom, were cured to constant weight at 140 F. and then tested, using two different testing molds with diameters of 2.000 and 2.021 in. The average strength values were 8500 psi. for the smaller diameter mold and 6260 psi. for the larger diameter.

TABLE VIII.—B.P.C.R. COMMITTEE COMPARISON TESTS.
South Carolina Soil[a]

Laboratory	Molding Moisture, per cent	Molded Dry Soil Density		Absorption		Expansion		Strength	
		Range, lb. per cu. ft.	Average, lb. per cu. ft.,	Range, per cent	Average, per cent	Range, per cent	Average, per cent	Range, psi.	Average, psi.
Bitumuls	6.05	121 to 122	122	1.0 to 1.5	1.3	3.0 to 3.0	3.0	1795 to 1925	1873
Barrett	5.94	121.2 to 121.7	121.5	1.0 to 1.1	1.0	3.6 to 4.2	4.0	1900 to 2050	1975
Esso	6.05	121.1 to 121.1	121.1	1.1 to 1.3	1.2	3.6 to 3.6	3.6	1900 to 2000	1950
Koppers	6.09	122.5 to 122.9	122.7	1.5 to 2.0	1.7	3.0 to 3.0	3.0	1750 to 1950	1867
Average	6.03		121.8		1.3		3.4		1916
Maximum Difference	0.15		1.6		0.7		1.0		108
Maximum deviation from average, per cent	1.5		0.7		30.8		17.6		3.1

[a] Mix composition when molded—per cent by weight:

South Carolina soil—dry	100.0
RT-3 Tar	6.0
Moisture	6.0

TABLE IX.—B.P.C.R. COMMITTEE COMPARISON TESTS.
San Francisco Soil[a]

Laboratory	Molding Moisture, per cent	Molded Dry Soil Density		Absorption		Expansion		Strength	
		Range, lb. per cu. ft.	Average, lb. per cu. ft.	Range, per cent	Average, per cent	Range, per cent	Average, per cent	Range, psi.	Average, psi.
Bitumuls	8.02	121 to 121	121	4.0 to 4.1	4.1	4.6 to 6.1	5.6	7080 to 7130	7112
Barrett	8.13	120.5 to 120.9	120.6	4.4 to 4.5	4.5	8.5 to 8.5	8.5	6380 to 6575	6468
Esso	8.12	118.9 to 119.0	119.0	4.3 to 4.5	4.4	7.9 to 8.5	8.1	7150 to 7250	7183
Koppers	7.87	118.8 to 119.2	119.0	3.3 to 4.1	3.7	6.1 to 6.1	6.1	7000 to 7450	7175
Average	8.04		119.9		4.2		7.1		6985
Maximum difference	0.26		2.0		0.8		2.9		715
Maximum deviation from average, per cent	2.1		0.9		11.9		21.1		7.4

[a] Mix composition when molded, per cent by weight:

San Francisco soil—dry	100.0
Emulsified asphalt—dry	6.2
Moisture	8.0

Table VI also shows the effect of a slight variation in the internal diameter of testing molds, the tests having been run in three different laboratories. Each laboratory made the same mixture, molded and cured the specimens at 140 F. to constant weight, then tested them for strength by the B.P.C.R. procedure. The internal diameters of the three test cylinders used were 2.000, 2.019, and 2.029 in. The average strength values were 6400, 4388, and 4100 psi. respectively. The data in Tables V and VI are plotted in Fig. 6. This plot indicates that for strength values of 4000 psi. or more the maximum allowable variation in the inside diameter of the testing cylinder (±0.001 in.) would affect the B.P.C.R. strength value by slightly over 100 lb., but for lower strength values this variation is undoubtedly less.

TABLE X.—B.P.C.R. COMMITTEE COMPARISON TESTS.
New Jersey soil[a]

Laboratory	Molding Moisture, per cent	Molded Dry Soil Density		Absorption		Expansion		Strength	
		Range, lb. per cu. ft.	Average, lb. per cu. ft.	Range, per cent	Average, per cent	Range, per cent	Average, per cent	Range, psi.	Average, psi.
Bitumuls	6.10	114 to 115	115	2.6 to 2.9	2.8	4.5 to 6.1	5.6	910 to 945	930
Esso	6.08	115.0 to 115.2	115.1	2.5 to 2.6	2.6	4.8 to 4.8	4.8	875 to 900	892
Koppers	5.89	115.9 to 116.1	116.0	2.3 to 2.6	2.4	4.6 to 4.6	4.6	1050 to 1065	1058
Average	6.01		115.4		2.6		5.0		960
Maximum difference	0.24		1.0		0.4		1.0		128
Maximum deviation from average, per cent	2.5		0.5		7.7		12.0		10.2

[a] Mix composition when molded, per cent by weight:

New Jersey soil—dry	100.0
MC-2 cutback asphalt	9.0
Moisture	6.0

TABLE XI.—SUMMARY OF DATA FOR B.P.C.R. COMMITTEE COMPARISON TESTS.

Soil	South Carolina	San Francisco	New Jersey
Mixture			
Water, weight, per cent	6	8	6
Binder, weight, per cent	RT-3 Tar 6	Asphalt Emulsion 11 (6.2 asphalt).	MC-2 Cutback 9
Molded Density (dry soil basis)			
Average value, lb. per cu. ft.	121.8	119.9	115.4
Maximum difference, lb. per cu. ft.	1.6	2.0	1.0
Maximum deviation from average, per cent	0.7	0.9	0.5
Absorption			
Average value, weight, per cent	1.3	4.2	2.6
Maximum difference, weight, per cent	0.7	0.8	0.4
Maximum deviation from average, per cent	30.8	11.9	0.8
Expansion			
Average value, volume, per cent	3.4	7.1	5.0
Maximum difference, volume, per cent	1.0	2.9	1.0
Maximum deviation from average, per cent	17.6	21.1	12.0
Strength			
Average value, psi	1916	6985	960
Maximum difference, psi.	108	715	128
Maximum deviation from average, per cent	3.1	7.4	10.2

Length of Time Required for Test.—In Table VII are listed the various steps required in the performance of the B.P.C.R. test method. Note that for tar and liquid asphalt-treated soils, the usual time is 9 days with an additional 2 days if cured specimens are desired for test. In testing emulsified asphalt-treated soils, seventeen days are usually required. The difference in time is, of course, due to the drying period when the wet mixture is air-dried to the specified molding moisture content, and to the oven-curing period during which 80 to 90 per cent of the molding moisture is removed.

Reproducibility of Results. — Tables VIII, IX, X, and XI show the range in values of B.P.C.R. test results reported by the cooperating laboratories, using the three different soils. They indicate that the specified tolerance limits for molding moisture content (±0.15 per cent) can easily be met, that close agreement in molded density values can be obtained, and that absorption, expansion, and strength values can be obtained that check very well with those determined by other laboratories. Table XI is a summary of the data contained in Tables VIII, IX, and X. It illustrates plainly the reproducibility of the B.P.C.R. test method.

SCOPE AND PRESENT STATUS

The B.P.C.R. Committee was formed originally to develop a method of test, that might be used by a federal, state, county or municipal authority, to evaluate bituminous stabilized soil mixtures recommended by various producers. As yet, there has been insufficient experience with the B.P.C.R. test method to establish minimum strength and maximum absorption and expansion values. Although the procedure might be developed into a design method, the present state of its development does not permit recommendation of its use for that purpose. The three soils used in this cooperative investigation were of the more or less cohesive type with which bituminous materials are admixed to impart resistance to softening action in the presence of moisture. The test method is intended primarily for use with soils of this type, rather than for granular noncohesive materials, such as sand, which are frequently classified as soil.

In the development of this test method particular care has been exercised to provide control features that eliminate many of the factors responsible for poor reproducibility of test results by different laboratories. The method of test is not too dissimilar from many now in use. Much of the required equipment is at present available in laboratories testing bituminous materials; and the other equipment can readily be fabricated in any well-equipped machine shop at a nominal cost.

The B.P.C.R. test method was submitted in 1944 to the Joint D-4, D-18 Subcommittee on Specifications and Tests for Stabilized Soils, and is now under their consideration. The method was published in the A.S.T.M. "Procedures for Testing Soils" (September 1944),[3] under the title: "Suggested Methods of Test for Water Absorption, Expansion, and Strength Characteristics of Soil-Bituminous Mixtures."

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[3] Issued as separate publication.

DISCUSSION

Mr. V. A. Endersby[1] (*by letter*).—*Concerning Considerations.*—(page 1418). a) is correct (*b*) is correct in principle, but why an extrusion test to furnish side support? Why not a triaxial or a bearing test?

Every engineer familiar with the fundamental mechanics of granular materials recognizes that the extrusion test does not report results in fundamental units of strength; that it is vitally affected by small variations in dimensions such as occur frequently through normal wear of equipment; and that it is more than anything else a measure of arching effects of the granular particles. Figure 3, for instance, shows that a 0.025-in. variation in the testing mold makes a difference of up to 50 per cent in the test results. Why introduce this unnecessary variable? The Public Roads Administration among others has changed to the use of compression tests for evaluating cohesive effects in aggregates, and in soils cohesive effects are the most important.

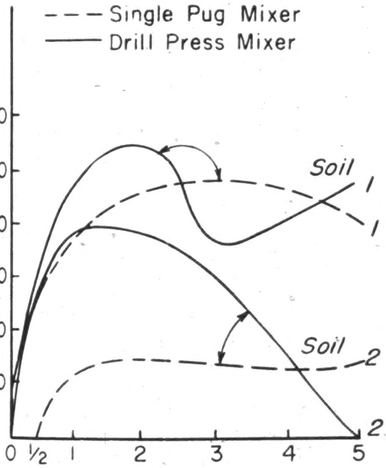

Fig. 7.—Influence of Mixer for Two Mixing Times.

Mixing Methods.—Benson and Becker[2] and Endersby[3] have shown that the degree of mixing is of vital importance to the results, and that this in turn can be vitally affected by the mixer used. It is possible that the three mixers specified in the subject test all have the same time-mixing properties; but it is more likely that the long mixing time of 5 min. produces an intimate mix which levels out the differences but does not

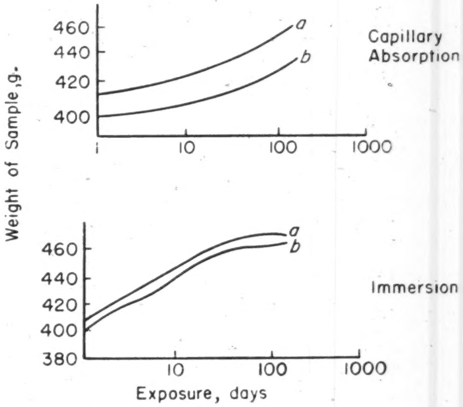

Fig. 8.—Influence of Curing on Water Resistance.

Curve a—Air-dried.
Curve b—140 F. oven curing.

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[1] Asphalt Dept., Shell Development Co., Emeryville, Calif.

[2] J. R. Benson and C. J. Becker, "Exploratory Research in Bituminous Soil Stabilization," *Proceedings*, Am. Assn. Paving Technologists, Vol. 13, p. 120 (1942).

[3] V. A. Endersby, "Fundamental Research in Bituminous Soil Stabilization," *Proceedings*, Highway Research Board, Vol. 22, p. 442 (1942).

correlate with what may be expected in road work. (See accompanying Fig. 7.)

Benson and Becker, and Endersby, through independent investigations, showed that the time of mixing has an optimum, which is likely to lie under one minute in high-clay soils; a mix which is highly resistant to water after mixing for one minute or less may be entirely ruined by mixing to 3, 4, or 5 min. (Fig. 7).

Moreover, the actions of liquid asphalts and of emulsions are entirely different; emulsions *require* thorough and intimate mixing; thus the mixing time and method specified will tend to show an untrue and undue degree of water resistance on the part of emulsion mixes as compared with liquid asphalts.

Compaction.—The use of a falling weight for tamping has been rejected by various investigators, among them Hveem,[4] because of its tendency toward breakage of aggregates.

This is in part true also of the 6000-lb. total load applied to a 2-in. specimen. Unit loads exceeding 1500 psi. tend to show breakage.

Curing.—Curing at high temperatures is well known to produce a permanently high water resistance as compared with curing at lower temperatures (Fig. 8).

Since curing at 140 F. is specified for emulsion, this is another point in which emulsion mixes will show unduly high results. Even if the alternative of high-temperature curing for liquid asphalts is used, the difference in favor of emulsions will remain until such additional curing has taken place as will eliminate all volatiles. Besides this, the thorough curing of the emulsion leaves a hard residue which acts as a binder in itself, lending a strength to the mix additional to that of the cohesive properties of the soil. This additional strength will not remain a property of the soil after long exposure to water unless the antistripping properties of the soil-emulsion combination are high.

The misleadingly high strength of emulsion mixes as produced by (*a*) excessive mixing and (*b*) high-temperature curing, is clearly shown in Table XI, where 1916 lb. for tar, 6985 lb. for emulsion, and 960 lb. for MC-2 cutback, are certainly not due to differences in the soil properties. This would have been more clearly shown had comparisons of the three treatments been made on the same soils.

Summary.—The objections to the test are:

1. Use of the extrusion test.
2. Mixing methods tending to give misleading results as to real water resistance and especially tending to show emulsion as highly superior to other treatments.
3. Curing times not in accordance with field conditions and tending to show the same misleading results on emulsion mixes.

Recommendations:

1. Substitution of a compression test or if desired, a triaxial test at a given lateral pressure, for the extrusion test.
2. Introduction of differential mixing times to determine mixing optima, and rigid specification of the mixer to be used.
3. Further investigation to determine the actual durability of the resistance to water action of emulsion mixes as compared with the others, when cured at temperatures properly correlated with field practice.

MR. W. S. HOUSEL[5] (*by letter*).—The paper presented by Messrs. Rhodes and Phelan makes available the results of an extremely comprehensive and painstaking investigation of the proposed method for evaluating soil-bituminous mixtures. The authors and the Bituminous Producers Cooperative Research Committee are to be commended for perseverance and continued effort over the trying period of the past several years in bringing such an investigation to a definite conclusion.

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[4] F. N. Hveem, Staff Materials and Research Engineer, Materials and Research Dept., Division of Highways, Sacramento, Calif.

[5] Associate Professor of Civil Engineering, University of Michigan, and Research Consultant, Michigan State Highway Dept., Ann Arbor, Mich.

It is understood that the proposed method is to be presented to the appropriate committees of A.S.T.M. and to be voted on in the near future. For that reason the writer wished to discuss the paper and present certain basic objections for consideration of the committees in the hope that these objections could be eliminated before the test is accepted by the Society.

In presenting these objections it is not the writer's intention to attempt to obstruct the sincere efforts of the committee to obtain a uniformly accepted method of evaluating soil-bituminous mixtures. It is the writer's objective to have written into the proposed test certain qualifications which would make it clear that the test is not to be accepted as a final measure of the performance characteristics of these mixtures under the conditions which regulate the design of paved surfaces. The acceptance of the test with the stamp of A.S.T.M. approval implies just that to the engineering profession at large. Until applicability as a design test has been demonstrated, acceptance should be qualified in order to encourage further research and development of a more fundamental method of evaluating stabilized mixtures. Otherwise the status given the test presently proposed may handicap further progress.

In the introductory portion of the paper the authors present the conclusions made by the committee upon which the subsequent investigation was apparently predicated. With the first and last of these conclusions the writer cannot agree.

In the first place, it is stated that "rigid and nonrigid base mixtures cannot be evaluated by the same test procedures because the two types are fundamentally different in design and performance." Taking a purely objective viewpoint, the writer cannot harmonize this statement with the fact that, regardless of whether the mixture is classified as rigid or nonrigid, it must in a paved surface be designed to carry the same loads, over the same subgrades, and under the same conditions of exposure and traffic. Failure to recognize these facts is a failure to be realistic, which can hardly be considered consistent with the principles of scientific research. The problem may be complex and our present knowledge inadequate, which can only mean that we must continue our efforts to develop a more realistic method of measuring performance and not accept without appropriate qualification the temporary expedient. Certainly research workers should eventually be able to find something which would simulate more closely the fairly obvious physical conditions of a load applied to a prepared soil base and supported by a soil subgrade.

In the last introductory conclusion cited in the paper it is stated that the "Bituminous stabilized soil mixtures preferably should be evaluated by some type of extrusion test wherein failure occurs due to excessive plastic flow of the mixture under test." While "failure" of the sample in the proposed extrusion test may be due to excessive plastic flow, no evidence is presented that failure in a paved surface is at all comparable to the conditions imposed in the test. The least that could be done in accepting the test would be to make sure that no busy and casually informed practicing engineer would rely on performance in the test to represent performance in the road.

Perhaps some constructive suggestions can be made by discussing the second introductory conclusion cited in the paper. This statement is that "When nonrigid base mixtures are subjected to any type of load-bearing or strength test, the test specimens should have some type of side support if reproducible results or indications of service behavior are to be obtained. . . ."

In this statement are revealed two apparent objectives of the proposed method of test: first, reproducibility of results, and, second, "indications of service behavior." In so far as reproducibility of results is concerned, it is the writer's opinion that lateral support of the sample is not necessary. A few years ago the writer conducted an extensive investigation of bituminous stabilized mixtures in which the stabilometer test or "triaxial compression" was used as a basis of evaluating the strength or stability of the mixtures. For purpose of simplification and economy in obtaining comparative values on a large number of different mixtures, the unconfined compression test was used, which actually is a special case of triaxial compression in which the lateral support is zero. Results of these tests were quite consistent and comparisons of different mixtures followed as definite a pattern throughout the investigation as could normally be expected in such mixtures. While extensive duplication of identical samples was not provided, there was every evidence of acceptable reproducibility. From this, the writer concludes that lateral support is not necessary for reproducibility of results, and, as a matter of fact, the work of the committee indicates the necessity for extreme accuracy in controlling dimensional effects involved in the high degree of confinement which the extrusion test includes. This in itself is evidence that the introduction of a high degree of lateral confining influence may be the source of poor, rather than good, reproducibility in the test procedure.

The desirability or necessity of some type of lateral support if the test is to be a measure of service behavior is a contention with which the writer can express wholehearted agreement. It would appear that the magnitude and character of the lateral support furnished is the crux of the situation in connection with devising a test which will simulate service behavior. In the writer's investigation, the failure to supply any lateral support can be properly criticized as being less favorable to nonrigid mixtures than the actual conditions encountered in the road surface. However, in favor of the stabilometer test it may be said that it introduces the possibility of regulating the lateral support through any desired range, making it possible to reproduce field conditions whenever they can be specified. In other words, this test provides flexibility in meeting field conditions, which so far as the writer knows is not available in any other type of test.

In the investigation by the committee it would appear to the writer that their proposed test goes to the other extreme, furnishing such a high degree of lateral support and confinement that the so-called strength or stability value completely masks any inherent weaknesses which these mixtures may possess under actual service conditions. In the paper, the stability values reported show a range of several thousands of pounds amounting to pressure intensities of the same order of magnitude as compared to normal tire pressures not exceeding 100 psi. It is the writer's belief that an acceptable test in so far as indications of service behavior are concerned lies somewhere between these two extremes. It is further contended that the stabilometer or triaxial compression test is the one which gives the most promise of being capable of adaptation to the requirements of a service performance test.

As stated previously in this discussion, the writer does not wish to obstruct the committee's earnest effort to present a test for use in evaluating soil-bituminous mixtures. As explained further in the discussion, the primary objection is to the test being identified as a strength or stability test. With these objections in mind, the writer would be perfectly willing to accept the proposed test as a temporary expedient if all reference to strength or stability were deleted. This could rather simply be done by changing the term "strength characteristics" to "extrusion value" in the title and statement of scope, and making corresponding changes in the discussion of the test assembly under "Apparatus," and in the description of the test itself in the concluding paragraph of the proposed test procedure.

To some, it might appear that the writer's objections are rather superficial, inasmuch as the objections can be eliminated by what might be even termed editorial corrections. However, it is believed that from the preceding discussion it can be appreciated that the objections are much more fundamental than that and involve principles which should not be compromised. As the matter stands, if the test were presented for acceptance under its present title and with the unavoidable implication that it provides "indications of service behavior," the writer would feel compelled to cast a negative vote and would likewise feel compelled to attempt to persuade others to take similar action.

MR. P. F. PHELAN (*author's closure by letter*).—When the paper, "Development of a Method of Test for Soil Bituminous Mixtures," which is actually a report of the work of the Bituminous Producers' Cooperative Research Committee, was reviewed at the June 1946, meeting of the Joint D-4, D-18 Subcommittee on Tests and Specifications for Stabilized Soils, a lively discussion followed. A number of questions were asked concerning the test procedure, and a number of objections to the proposed test method were raised. When the paper was presented, Mr. Housel made further comment. Since then, Mr. V. A. Endersby has presented further remarks concerning the test method. The more important comments made at the Joint D-4, D-18 meeting and those presented by Messrs. Housel and Endersby are treated in detail below:

Need for Test:

The need for a test of this kind is evident from the fact that the "Procedures for Testing Soils" published in 1944 contains fourteen methods of test proposed for use in testing soil-bituminous mixtures. In addition to the fourteen methods listed therein, there are other methods—such as the Texas Modified Bearing Value Test—and modifications and combinations of some of these tests that would possibly bring the total of the tests now in use for bituminous stabilized soil mixtures to at least twenty-five. A large number of these test methods are quite similar as to size of specimen, type and amount of compaction, and types of absorption and strength tests. In the interests of progress, efficiency, and economy, it is only logical that someone or some group should attempt to develop a mutually acceptable test method that might be used in referee cases.

The need for this type of test was voiced by the Civil Aeronautics Authority to members of the B.P.C.R. group over five year ago. In fact, that was the reason why the cooperative development of this method of test was begun.

Curing Specimens:

In the "Procedures for Testing Soils," all but one of the fourteen test methods proposed for use with soil bituminous mixtures require or allow curing of some kind, and all but one of these specify that the curing be carried out at 140 F. It appears, then, that the consensus of opinion of those who test and work with these mixtures is that curing of some type—either compulsory or optional—should be included in any test method, and that this curing should be done at 140 F.

In the scope of the test method, it is stated that "*unless otherwise specified*, soil emulsified asphalt specimens shall be tested in the *cured* state and soil asphalt and soil tar specimens shall be tested in the *uncured* state." Concerning the soil asphalt and soil tar specimens, it is stated in Section 8 (*c*) of the test method that, "if desired, additional specimens may be tested in the *cured* state and the results identified as such." In other words, it is required *unless otherwise specified* that the emulsified asphalt soil mixtures be cured prior to test, and recommended that the cutback asphalt soil and tar soil mixtures be tested in the uncured state. Provision is made, however, for testing cured specimens of soil asphalt and soil tar, if cured specimens are desired.

Difference in Degree of Curing—Emulsified Asphalt versus Cutback Asphalt and Tar:

When soil-water-emulsified asphalt specimens are cured at 140 F., all of the loss is moisture loss, so that a definite percentage of loss can be specified. When curing to a definite percentage of loss, any differences in the efficiencies of drying ovens are thus prevented from appreciably affecting the final test results for water absorption and strength.

However, if soil-water-cutback asphalt or soil-water-tar mixtures are cured at 140 F., the loss is not all moisture but is, in part, loss of volatiles from the bituminous material employed. Since this amount of volatile loss varies with the type and composition of the bituminous material, it would be difficult to specify a definite percentage of loss. Therefore, it was thought best to cure such specimens—if curing is desired—to constant weight. It is admitted that different ovens may cure to various degrees due to different efficiencies, but this objection could be overcome only by very complete specifications for the type, size, construction, and operation of ovens to be used for curing.

Use of Strength Test Results:

The strength test is included in the method only to assist in determining the optimum percentage of bituminous material for use with a given soil. The strength data are not intended for use in the design of subbase, base or pavement thickness.

Extrusion versus Strength Test:

Mr. Housel has suggested that the test method measures "extrusion value" rather than "strength" of the mixture under test. It is admitted to be a moot question as to whether the extrusion or punching shear method used in the proposed test is truly a measure of mixture strength applicable to stabilized soil road construction. However, there is no objection to stating that the "stability," "strength" or "load resistance" is determined by an extrusion or punching shear test.

B.P.C.R. Test versus Triaxial or Compression Test:

Objections have been raised to the use of an extrusion type of test. The punching shear test as employed in the proposed procedure expresses results which can be reduced to unit shear and are, it appears, as scientific as those obtained with a triaxial or a compression test. One of the features of the usual triaxial test is a so-called "cohesion" value which is calculated from the test results and generally bears little relation to the actual cohesion of a soil mixture as measured by simple tension tests.

The necessity for accurate dimensions of equipment, as indicated by the effect of a slight variation in the internal diameter of the testing mold (Fig. 3), merely emphasizes the need for maintaining the testing equipment in proper condition. This does not appear to be a valid criticism of the test method itself.

Differential Mixing Times to Determine Mixing Optima:

The "fluff-point" or optimum mixing time, suggested by Mr. Endersby, appears to be dependent upon the equipment, as indicated in Fig. 7 of his comments. With soil No. 1, the drill press mixer gives a maximum strength of 54 lb. in $1\frac{3}{4}$ min., whereas the single pug mixer reaches the optimum point in 3 min. with the mixture having a strength of 49 lb. With soil No. 2, the drill press mixer reaches the "fluff-point" at 1 min. with a strength of 40 lb.; whereas the single pug mixer gives optimum strength of 14 lb. in $1\frac{3}{4}$ min. and continues at that optimum strength up to a mixing time of about 5 min.

The main object in the development of the B.P.C.R. test procedure was to devise a method which would give results that could be reproduced by different laboratories. For this reason, definite mixing times based upon laboratory experience were selected and specified in the test method. The "fluff-point" or optimum point depends upon catching the optimum mixture "on the run." Certainly, any procedure based upon such a feature will encounter great difficulty in yielding reproducible results, let alone reproducing such "on the fly" laboratory results during field construction.

Aggregate Breakage During Compaction:

Since the test procedure was designed primarily for use with soils (passing the No. 4 sieve) with which bituminous materials are admixed to impart resistance to the softening action of moisture, it is not intended for use with gravel. The pre-compaction tamping—25 blows of a 1200-g. (2.5-lb.) tamper dropped from a height of 6 in. above the *bottom* of the specimen being formed—is intended only to orient the particles in the mixture, so as to obtain more uniform density in the specimen when it is finally compacted—under a 6000-lb. load held for 2 min. This pre-compaction tamping, then, is not intended primarily for compaction, as is true in the Proctor or Modified Proctor Methods, in which the 5.5-lb. rammer is dropped from a height of 12 or 24 in. above the *top* of the specimen being formed. Thus, the risk of crushing the soil particles when following this procedure is practically nil.

For clean gravels (for which this test is not intended) excessive compaction loading will produce crushing. There does not seem to be any general agreement as to the maximum permissible loading in such cases. It appears that this value must be determined for each type of gravel.

Validity of Test Results:

The test procedure does not attempt to advocate that a given soil bituminous mixture shall or shall not be cured prior to test; it merely outlines the operations for either case as the problem may require. Nor has the procedure been devised to indicate a preference for or advantage of any one bituminous material over another for use in stabilized soil mixtures, but merely to give a test method best suited to each product—whether cutback asphalt, emulsified asphalt, or tar. The effects of mixing, curing, water immersion, and strength testing were each considered in detail and thoroughly explored in the development of the test method. The matter of personal opinion concerning the various steps in the test procedure was thus eliminated.

As mentioned in the paper, the proposed method of test is quite similar to many already in use. In the development of this method, particular care was taken to provide control features that eliminate many of the factors in present test methods that are responsible for poor reproducibility of results by different laboratories. The committee has shown that with this test method, reproducible results can be obtained.

Present Status:

The proposed test method was published in A.S.T.M. "Procedures for Testing Soils" (September, 1944), under the title "Suggested Methods of Test for Water Absorption, Expansion and Strength Characteristics of Soil Bituminous Mixtures." It has also been published as Appendix B in the Highway Research Board Current Road Problems Bulletin No. 12, "Soil Bituminous Roads." It is now under consideration by A.S.T.M. Joint D-4, D-18 Subcommittee on Tests and Specifications for Stabilized Soils.

A number of editorial revisions in the proposed method have been suggested by members of the Joint Subcommittee. These include changing the title, rewording the "Scope," and substituting some other term for the word "strength" throughout the method. Thus far, no changes have been suggested regarding specimen size, molding and testing equipment, preparation of mixtures, formation of specimens, or procedures for curing and testing for absorption, expansion, and strength characteristics.

A METHOD OF DETERMINING THE PARTICLE SIZE OF COLLOIDS FROM SEDIMENTATION DATA OBTAINED WITH THE SHARPLES SUPERCENTRIFUGE[1]

BY SERGE A. LOUKOMSKY[2] AND S. JAMES O'BRIEN[2]

There are in the literature several studies which indicate the applicability of the Sharples Supercentrifuge for the estimation of the particle size and the fractionation of colloidal systems. In most of these (**1, 2, 3, 4, 5, 6, 7**)[3] little consideration was given to the mathematical theory underlying the sedimentation of particles in this instrument. Hauser and Reed (**8**), however, have developed a theory explaining the sedimentation of a particle in the Supercentrifuge and the relationship of the sedimentation data to the particle size distribution of the sedimented material. Their method of applying this theory was left in a rather complicated form. In later work by Hauser and Lynn (**9**) and by Hauser and Schachman (**10**), the method was simplified, but certain restrictions were introduced that limit its applicability to systems having a narrow range of particle sizes and to certain experimental conditions. It is the purpose of this paper to suggest a method based on the work of Hauser and Reed which will permit a relatively easy calculation of the particle-size distribution of a system from sedimentation data such as those shown in Fig. 1, without confining the procedure to any definite set of experimental conditions. The data in Fig. 1 represent the result of a typical sedimentation of an aqueous pigment suspension giving the weight of sediment collected in each centimeter interval along the length of the centrifuge bowl. However, before discussing this curve further, it may be well to review briefly the operation of the Supercentrifuge and the equation for the trajectory of a particle as given in the papers of Hauser and coworkers (**8, 9, 10**).

In Fig. 2 is shown a schematic cross-section of the bowl of the Supercentrifuge. The suspension is fed into the rapidly rotating bowl at the bottom; it passes two straightening vanes at right angles to each other and flows up the sides of the bowl in a film forming a cylindrical ring of radii R_1 and R_2. R_1 is the radius of the column of air through the center of the bowl and R_2 is the inside radius of the bowl.

Since a particle has a velocity component parallel to the axis of rotation due to the flow of liquid through the bowl, and a velocity component at right angles to the bowl due to centrifugal force, the path of the particle is curved as indicated in the figure. The point at which a particle of a given size strikes the wall of the bowl depends upon the distance from the axis of rotation X_w, where the particle enters the bowl. The nearer the axis of rotation that it enters the bowl the higher it will strike; the further away from the axis of rota-

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[1] Presented at a technical session sponsored by Committee D-1 on Paint, Varnish, Lacquer and Related Products, held in Buffalo, N. Y., June 26, 1946.

[2] Assistant Chief Physicist and Assistant Chief Chemist, respectively, Calco Chemical Division, Am. Cyanamid Co., Bound Brook, N. J.

[3] The boldface numbers in parentheses refer to the references appended to this paper, see p. 1450.

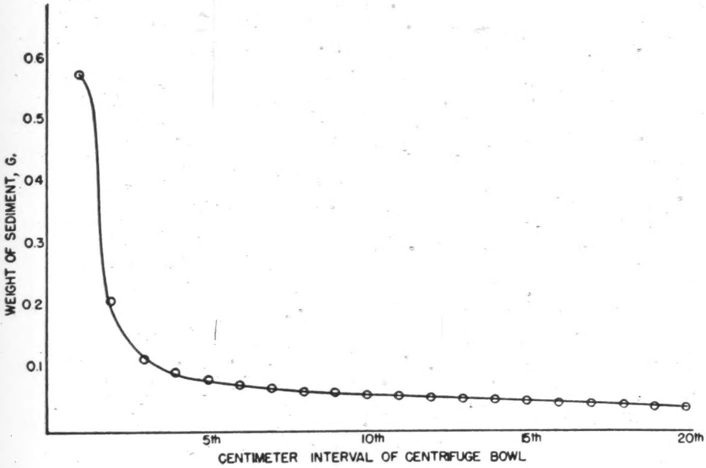

FIG. 1.—A Plot of the Weight of Sediment in Each Centimeter Interval of the Centrifuge Bowl in the Centrifugation of an Aqueous Pigment Dispersion.

tion the lower it will strike. This may be stated mathematically by the equation

$$X_0 = f(y, D) \qquad \text{(1)}$$

following the convention and notation of Hauser and Reed (8).

From the hydrodynamics involved in the flow of the cylindrical ring of liquid through the centrifuge and the law of Stokes as applied to a centrifugal field, Eq. 1 may be further developed to give

$$y = \frac{18\eta\alpha D^2}{\omega^2 \Delta\rho} \left[\frac{R_2^2}{4}\ln\frac{R_2}{X_0} - \left(\frac{R_1^2}{4}\ln\frac{R_2^2}{X_0}\right) + \frac{X_0^2 - R_2^2}{8}\right] \qquad \text{(2)}$$

where:

y = the distance parallel to the axis of rotation traveled by a particle starting from $y = 0$; $X = X_o$,

ω = angular velocity of the Centrifuge (radians per second),

X_o = the distance from the axis of rotation (centimeters),

R_1 = the radius of the column of air through the center of the bowl, (centimeters),

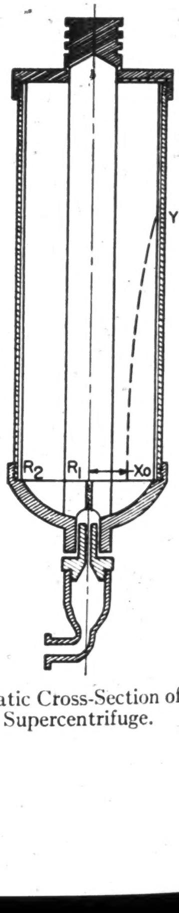

FIG. 2.—Schematic Cross-Section of the Sharples Supercentrifuge.

R_2 = the inside radius of the bowl minus the thickness of the liner (centimeters),

D = the equivalent spherical diameter of the particle (centimeters),

η = the coefficient of viscosity of the dispersion fluid (poises),

$\Delta\rho$ = the difference in density between the disperse and continuous phases (grams per cubic centimeter), and

α = a constant depending on the volumetric rate of flow of liquid, Q, through the centrifuge and the dimensions of the centrifuge. It is further defined by

$$\alpha = \frac{8Q}{3R_1^4 + R_2^4 + 4R_1^4 \ln R_2/R_1 - 4R_1^2R_2^2} \quad \ldots (3)$$

Except for the evaluation of α in terms of the average rate of flow of the suspension through the centrifuge, this equation is the same as that of Hauser and Reed (8) for the trajectory of a particle. It should be emphasized that in deriving this equation, streamline flow parallel to the axis of rotation is assumed and this may not be strictly true, especially at the ends of the bowl.

The problem at this stage is the application of Eq. 2 to the determination of the particle-size distribution of the sedimented material, avoiding the difficulties of the method of Hauser and Reed and the restrictions of Hauser and Lynn and Hauser and Schachman. Briefly, the method of Hauser and Reed is difficult because it involves the solution of sets of n simultaneous equations in n unknowns in a method of successive approximations. The restrictions introduced in the later work are "that the system under consideration be well defined (particle sizes covering but a small range) and that passage through the centrifuge be at an appropriate rate" (9). With these restrictions, Hauser and coworkers consider that Eq. 1 is simplified to

$$y = f(X_0) \quad \ldots\ldots\ldots\ldots (4)$$

which states that particles of only one size reach the point y on the wall of the centrifuge. While the calculations are thus simplified, no data obtained in this laboratory in the sedimentation of a large number of pigment dispersions under all sorts of experimental conditions have been amenable to this treatment. Consequently, a method was sought which would be in conformity with the kind of sedimentation data obtained in our studies and which would be simpler and more direct than that of Hauser and Reed. A stepwise procedure was developed by taking into consideration the amount of sediment in each small interval along the wall of the bowl and by dealing with a single particle size at a time.

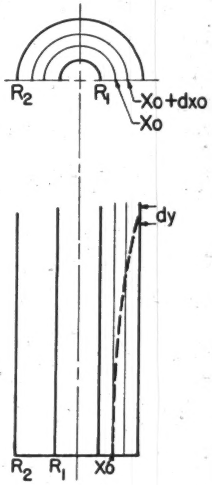

FIG. 3.—Sedimentation of a Particle in the Supercentrifuge.

DENSITY OF SEDIMENT

The first step in the development of a stepwise method is, as indicated above, the calculation of the density of the sediment, that is, the weight of sediment per unit of length along the length of the centrifuge bowl. If particles of only one size are considered, it will be evident from Fig. 3 that all particles of this size entering the centrifuge in a ring bounded by the circles X_o and $X_o + dX_o$ will be deposited on the wall of the bowl at some interval between y and $y + dy$, the value of y depending on that of X_o in accordance with Eq. 2.

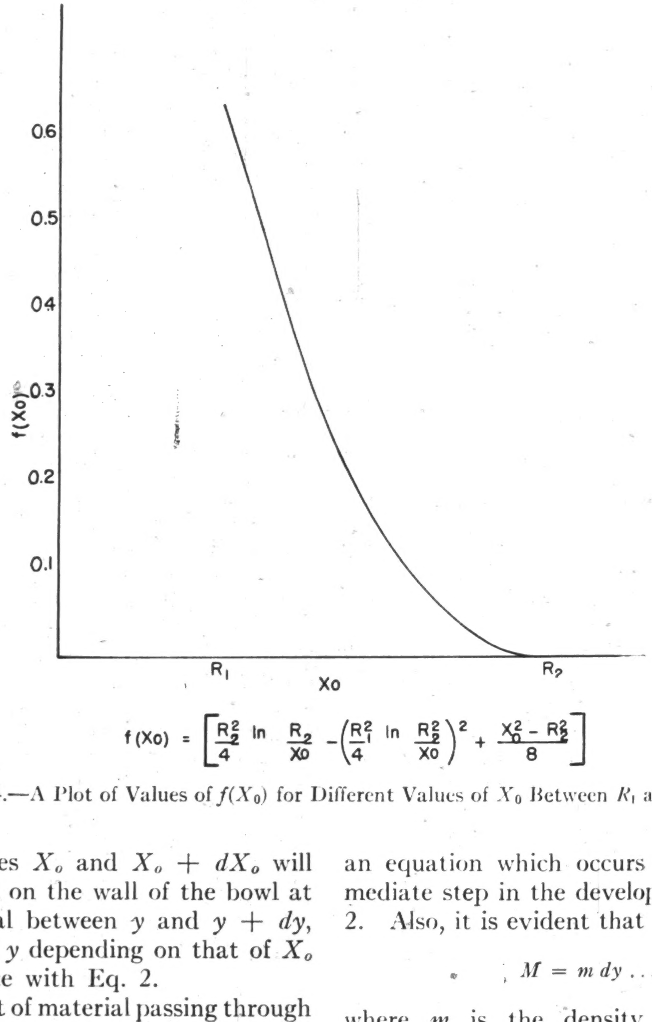

FIG. 4.—A Plot of Values of $f(X_0)$ for Different Values of X_0 Between R_1 and R_2.

The weight of material passing through such a ring will be given by

$$M = Cut\, 2\pi X_0 \cdot dX_0 \,\ldots\ldots\ldots \quad (5)$$

where M is the weight of the particles, C is the concentration of the monodisperse suspension (grams per cubic centimeter), t is the time required for the sedimentation, and u is the local velocity of the suspension through the bowl, which is given by

$$u = \frac{dy}{dt} = \alpha\left(\frac{R_2^2 - X_0^2}{4} + \frac{R_1^2}{2} \ln \frac{X_0}{R_2}\right) \quad (6)$$

an equation which occurs as an intermediate step in the development of Eq. 2. Also, it is evident that

$$M = m\, dy \,\ldots\ldots\ldots\ldots \quad (7$$

where m is the density (grams per centimeter) of the deposit on the bowl between y and $y + dy$. Combining Eqs. 2, 5, 6, and 7 leads to

$$m = (2\pi Cl\alpha\omega^2 \Delta\rho D^2/18\eta) X_0^2 \ldots \quad (8$$

That is, for particles of uniform size and for a definite set of experimental conditions, the density of the deposit at any point along the length of the bowl is directly proportional to the square of the distance from the axis of rotation where the particles enter the centrifuge. The stepwise method is then developed using Eqs. 2 and 8.

STEPWISE METHOD OF APPROXIMATION

For particles of a single particle size Eqs. 2 and 8 may be written as

$$y = k_2 f(X_0) \qquad \text{(2a)}$$

$$m = k_3\, Ct\, X_0^2 \qquad \text{(8a)}$$

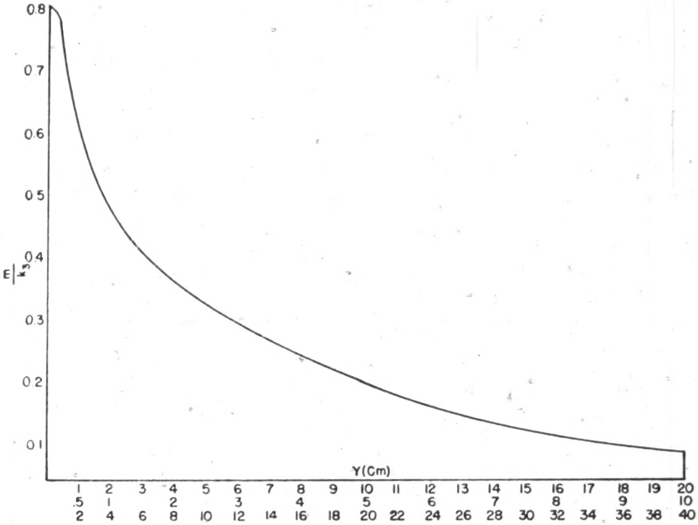

FIG. 5.—A Plot of the Density (in Grams per Cubic Centimeter) of the Sediment on the Bowl of the Centrifuge Against the Distance from the Bowl Entrance.

Since a relationship between y and m is desired, a plot of $f(X_o)$ against X_o is required. Such a plot is shown in Fig. 4, using the values of $R_1 = 0.734$ cm., and $R_2 = 2.222$ cm., for the centrifuge bowl used in our work. From Fig. 3, it is apparent that if a material of uniform particle size is completely sedimented within the bowl, those particles reaching the maximum value of y enter the centrifuge at $X_o = R_1$, and the density of the deposit at this point will be given by

$$m = k_3\, CtR_1^2 = k_2\, Ct\, (0.734)^2$$

The density of the deposit at lower values of y is obtained by evaluating k_2 in Eq. 2a and obtaining the corresponding value of X_o from the plot of $f(X_o)$ against X_o (Fig. 4). This is illustrated as follows:
when $y_{\text{max.}} = 20$ cm., then

$$k_2 = \frac{20}{f(X_0)_{X_0 = R_1}} = \frac{20}{0.660} = 30.3;$$

for $y = 10$ cm.

$$f(X_0) = 10/30.3 = 0.333$$

X_o is found from Fig. 4 to be 1.120 cm. and the density at $y = 10$ cm. of the deposit of particles which are completely sedimented at $y = 20$ cm. will be

$$m = k_3\, Ct\, (1.120)^2$$

Figure 5 shows how the density of the sediment varies with y for a material of uniform size which is completely sedimented at $y = 20$ cm. The calculations were made using $C = 0.005$ g. per cubic centimeter, $t = 333$ sec. From the form of Eqs. 2a and 8a, it is

TABLE I.—RATIO OF THE WEIGHT OF SEDIMENT OF UNIFORM SIZE IN EACH CENTIMETER TO THE WEIGHT IN THE LAST CENTIMETER FOR VARIOUS PARTICLE SIZES.

Centimeter Interval	Ratio of Weight in Each Centimeter to the Weight in the Last Centimeter when the Last Centimeter is:																				Centimeter Interval
	19 to 20	18 to 19	17 to 18	16 to 17	15 to 16	14 to 15	1 3 to 4	12 to 13	11 to 12	10 to 11	9 to 10	8 to 9	7 to 8	6 to 7	5 to 6	4 to 5	3 to 4	2 to 3	1 to 2	0 to 1	
19 to 20	1.00																				19 to 20
18 to 19	1.07	1.00																			18 to 19
17 to 18	1.16	1.08	1.00																		17 to 18
16 to 17	1.28	1.17	1.09	1.00																	16 to 17
15 to 16	1.40	1.29	1.18	1.11	1.00																15 to 16
14 to 15	1.53	1.41	1.30	1.21	1.10	1.00															14 to 15
13 to 14	1.66	1.55	1.48	1.33	1.23	1.11	1.00														13 to 14
12 to 13	1.78	1.69	1.58	1.50	1.36	1.24	1.11	1.00													12 to 13
11 to 12	1.92	1.81	1.71	1.64	1.51	1.36	1.26	1.12	1.00												11 to 12
10 to 11	2.12	1.99	1.86	1.78	1.67	1.54	1.42	1.29	1.13	1.00											10 to 11
9 to 10	2.34	2.21	2.09	2.00	1.83	1.69	1.59	1.46	1.32	1.13	1.00										9 to 10
8 to 9	2.57	2.45	2.30	2.20	2.16	1.89	1.77	1.70	1.47	1.32	1.18	1.00									8 to 9
7 to 8	2.83	2.69	2.55	2.45	2.31	2.16	2.02	1.86	1.72	1.47	1.39	1.20	1.00								7 to 8
6 to 7	3.16	3.09	2.85	2.77	2.60	2.42	2.30	2.16	1.98	1.72	1.63	1.46	1.22	1.00							6 to 7
5 to 6	3.55	3.39	3.18	3.07	2.97	2.86	2.62	2.49	2.33	1.98	1.93	1.72	1.51	1.26	1.00						5 to 6
4 to 5	3.95	3.80	3.74	3.54	3.44	3.26	3.09	2.94	2.80	2.33	2.31	2.11	1.82	1.65	1.33	1.00					4 to 5
3 to 4	4.46	4.33	4.24	4.15	3.95	3.78	3.62	3.44	3.28	2.80	2.86	2.63	2.33	2.05	1.73	1.39	1.00				3 to 4
2 to 3	5.02	4.89	4.80	4.77	4.57	4.39	4.28	4.13	3.97	3.28	3.54	3.35	3.04	2.72	2.38	1.95	1.52	1.00			2 to 3
1 to 2	5.61	5.58	5.55	5.51	5.32	5.16	5.07	5.00	4.89	3.97	4.48	4.31	4.02	3.74	3.40	2.93	2.44	1.76	1.00		1 to 2
0 to 1	7.95	7.77	7.63	7.54	7.44	7.23	7.13	7.00	6.78	4.89	6.50	6.33	6.01	5.77	5.49	5.06	4.55	3.85	2.85	1.00	0 to 1
Total	56.39	55.29	49.70	47.70	44.56	41.19	38.34	36.58	32.66	26.89	26.82	24.11	20.95	18.19	15.23	12.33	9.51	6.61	3.85	1.00	Total

obvious that a similar plot for material of any other size can be obtained from Fig. 5 simply by changing the coordinates as is illustrated with the second set of abscissae given in the figure.

In practice it is convenient to deal with the deposit in intervals of, say, 1 cm. rather than with the deposit at a point. Considering Fig. 5 again, the area under the curve is proportional to the total weight of material of a uniform size. The area under the curve between any two values of y is proportional to the weight of material of this size collected in that interval of the centrifuge bowl. The family of curves that may be obtained then are best summarized in a form such as that given in Table I, where the ratios of the area under the curve at each centimeter interval to the area under the last centimeter interval for material being completely sedimented in each centimeter interval of the bowl are tabulated.

TABLE II.—CALCULATION OF PARTICLE SIZE FROM SEDIMENTATION DATA WHEN MATERIAL IS COMPLETELY SEDIMENTED IN CENTRIFUGE BOWL.

Centimeter Interval	Gross Sediment, g.	19 to 20 cm. Sediment	Residue	9 to 10 cm. Sediment	Residue
19 to 20	0.0500	0.0500	...	...	...
18 to 19	0.0535	0.0535	...	...	...
17 to 18	0.0580	0.0580	...	...	...
16 to 17	0.0640	0.0640	...	...	...
15 to 16	0.0700	0.0700	...	...	...
14 to 15	0.0765	0.0765	...	...	...
13 to 14	0.0830	0.0830	...	...	...
12 to 13	0.0890	0.0890	...	...	...
11 to 12	0.0960	0.0960	...	...	...
10 to 11	0.1095	0.1095	...	...	...
9 to 10	0.1670	0.1170	0.0500	0.0500	...
8 to 9	0.1875	0.1285	0.0590	0.0590	...
7 to 8	0.2110	0.1415	0.0695	0.0695	...
6 to 7	0.2395	0.1580	0.0815	0.0815	...
5 to 6	0.2740	0.1775	0.0965	0.0965	...
4 to 5	0.3130	0.1975	0.1155	0.1155	...
3 to 4	0.3660	0.2230	0.1430	0.1430	...
2 to 3	0.4300	0.2530	0.1770	0.1770	...
1 to 2	0.5045	0.2805	0.2240	0.2240	...
0 to 1	0.7225	0.3975	0.3250	0.3250	...
Total	4.1645	2.8240	1.3410	1.3410	

APPLICATION OF METHOD WHEN OVERFLOW IS NEGLIGIBLE

The use of this method is illustrated by means of the following simple example of the sedimentation of a material, the weight of which is divided between two particle sizes, both being completely sedimented in the bowl. The sedimentation data obtained in such a centrifugation are given in the second column of Table II which gives the weight of the sediment collected in each centimeter interval along the wall of the bowl. The third column gives the weight in each centimeter interval of material of that size completely sedimented out in the 20th centimeter interval. It is calculated from the weight in the last centimeter interval and the ratios given in Table I for the 20th centimeter interval. The weight of the remaining material is given in the fourth column of this table. Since there is no residue beyond the 10th centimeter, the corresponding particle sizes are absent. The fourth column contains the weight of material of that size which is completely sedimented out in the 10th centimeter. This is calculated from the residue in the 10th centimeter and the ratios given in the corresponding column of Table I. The particle sizes of the two fractions are then calculated from the values of $y_{\text{max.}}$ (that is, 20 cm. and 10 cm.) using Eq. 2 in the form

$$y_{\text{max.}} = \frac{18\eta\alpha}{\omega^2 \Delta\rho D^2} \left[\frac{R_2^2}{4} \ln \frac{R_2}{R_1} - \frac{R_1^2}{4} \ln \frac{R_2^2}{R_1} + \frac{R_1^2 - R_2^2}{8} \right] \quad \text{(2b)}$$

or using the bowl constants for the centrifuge employed in this work

$$y_{\text{max.}} = \frac{1.92 Q\eta}{\omega^2 \Delta\rho D^2} \quad \text{(2c)}$$

While the numerical examples given above have included the last centimeter intervals, it should be emphasized that data taken from the sediment at the ends of the bowl are likely to be untrustworthy because of the possibility of turbulence. In practice, the deposit in the last two centimeters is included with the overflow. Some error will be introduced into the whole distribution by the necessity of including the sediment from the first 2 cm., but most of the uncertainty will show up with the largest particle sizes, that is, those completely sedimented in the first 2 cm.

APPLICATION OF METHOD WHEN OVERFLOW IS NOT NEGLIGIBLE

If the disperse phase is not completely sedimented in the bowl but passes partly into the overflow, the method is not as satisfactory. Yet, even in this case a fairly good approximation can be made. This is done by considering that the particle size of the material in the overflow is the same as that sedimented in one of the upper centimeters, for example, the 18th centimeter of the bowl. First, however, it is necessary to calculate the length the bowl required for complete settling of material of a single particle size, a certain weight of which is found in the 18th centimeter. The third

TABLE III.—CALCULATION OF PARTICLE SIZE FROM SEDIMENTATION DATA WHEN THE WEIGHT OF MATERIAL IN THE OVERFLOW IS SIGNIFICANT.

Centimeter Interval	Sediment	Ratio $\frac{m_y}{m_{18}}$	Overflow Sediment	Residue	9 to 10 Sediment	Residue
Overflow	0.194	...	0.194	...	...	...
19 to 20	0.008	...	0.008	...	...	...
18 to 19	0.009	...	0.009	...	...	...
17 to 18	0.010	1.00	0.010	...	...	...
16 to 17	0.010	1.04	0.010	...	...	...
15 to 16	0.011	1.09	0.011	...	...	...
14 to 15	0.011	1.13	0.011	...	...	...
13 to 14	0.012	1.17	0.012	...	...	...
12 to 13	0.012	1.23	0.012	...	...	...
11 to 12	0.013	1.29	0.013	...	...	...
10 to 11	0.013	1.33	0.013	...	...	...
9 to 10	0.114	1.39	0.014	0.100	0.100	...
8 to 9	0.132	1.44	0.014	0.118	0.118	...
7 to 8	0.154	1.52	0.015	0.139	0.139	...
6 to 7	0.179	1.59	0.016	0.163	0.163	...
5 to 6	0.210	1.66	0.017	0.193	0.193	...
4 to 5	0.249	1.75	0.018	0.231	0.231	...
3 to 4	0.305	1.86	0.019	0.286	0.286	...
2 to 3	0.374	2.00	0.020	0.354	0.354	...
1 to 2	0.471	2.26	0.023	0.448	0.448	...
0 to 1	0.678	2.78	0.028	0.650	0.650	...
Total	3.156	...	0.474	2.682	2.682	

$$\text{Equivalent overflow} = \frac{0.194}{10} \times 100 = 1.94.$$

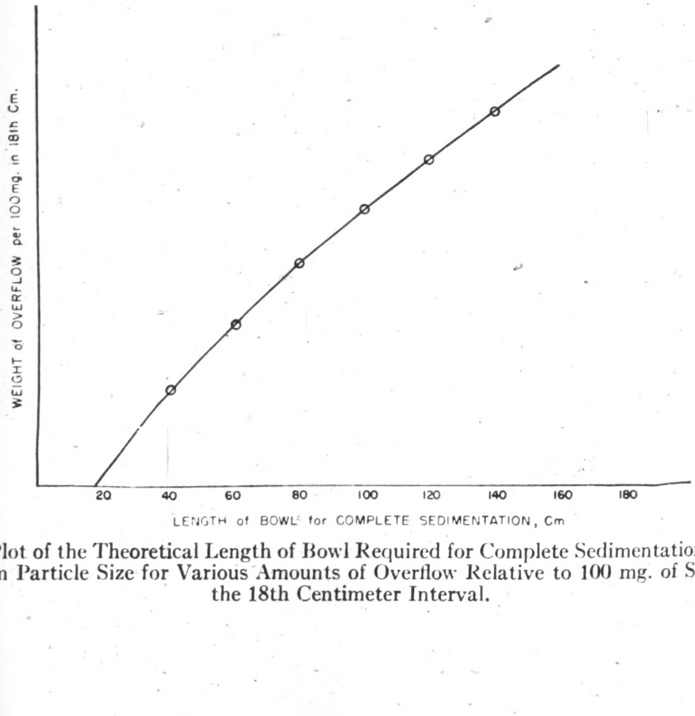

FIG. 6.—A Plot of the Theoretical Length of Bowl Required for Complete Sedimentation of Overflow of Uniform Particle Size for Various Amounts of Overflow Relative to 100 mg. of Sediment in the 18th Centimeter Interval.

set of abscissae in Fig. 5 indicates the relationship between the sediment of uniform size in the 18th centimeter and that in the overflow if the particles are completely sedimented in a bowl 40 cm. long. It is possible then to make a plot of the weight of material in the overflow against the length of bowl required to settle the particles completely. This is illustrated in Fig. 6, where the weight of overflow relative to 100 mg. of sediment in the 18th centimeter is plotted against the length of the bowl required for complete sedimentation.

The treatment of the data when the amount of the material in the overflow is significant is illustrated in Table III for a material that is made up of two particle sizes, one of which is completely sedimented in the bowl, a part of the other being carried into the overflow.

The total weight of sediment is given in the first column. From the weight of material in the overflow, the weight of the sediment in the 18th centimeter and Fig. 5, the length of bowl that would be required for the complete sedimentation of the "fines" is calculated. In order to use Fig. 5, which is based on 100 mg. of sediment in the 18th centimeter, it is

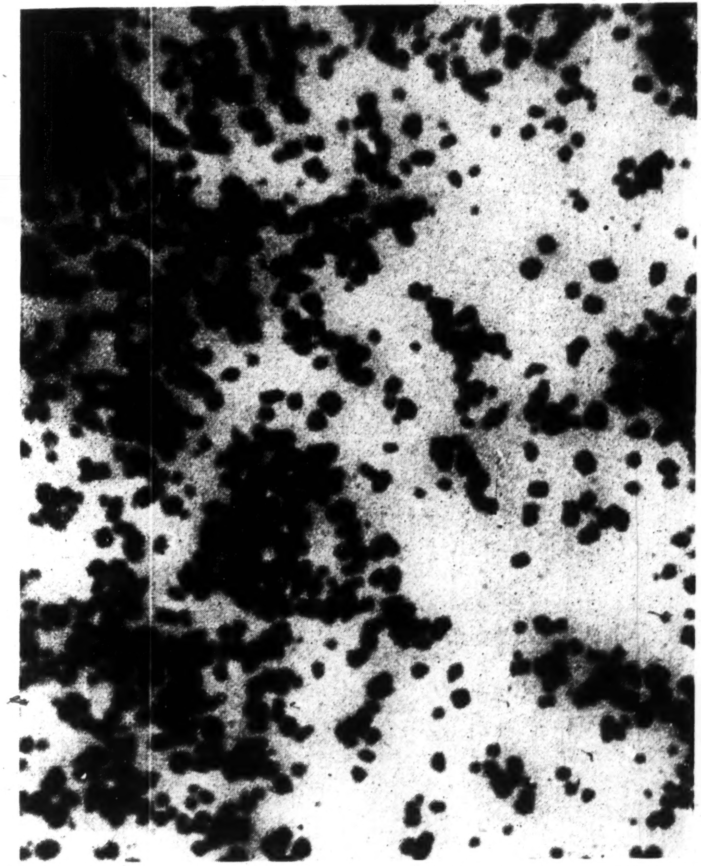

FIG. 7.—Electronmicrograph of Iron Blue After Grinding into Linseed Oil (× 16,000).

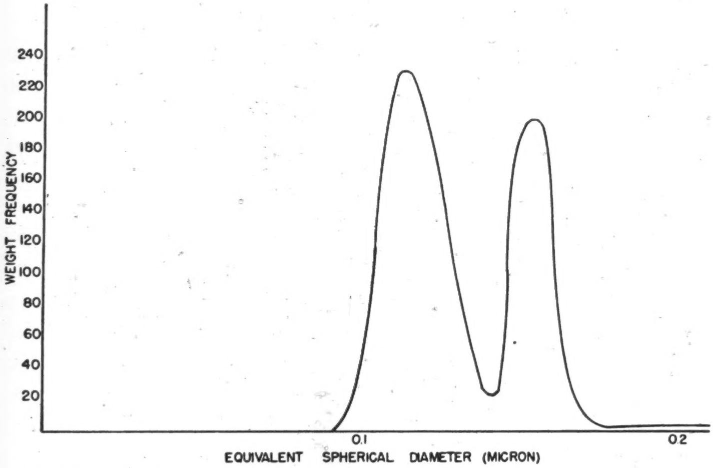

FIG. 8.—Particle-Size Distribution Obtained by Sedimentation in the Supercentrifuge for Iron Blue After Grinding into Linseed Oil and Diluting with Xylene.

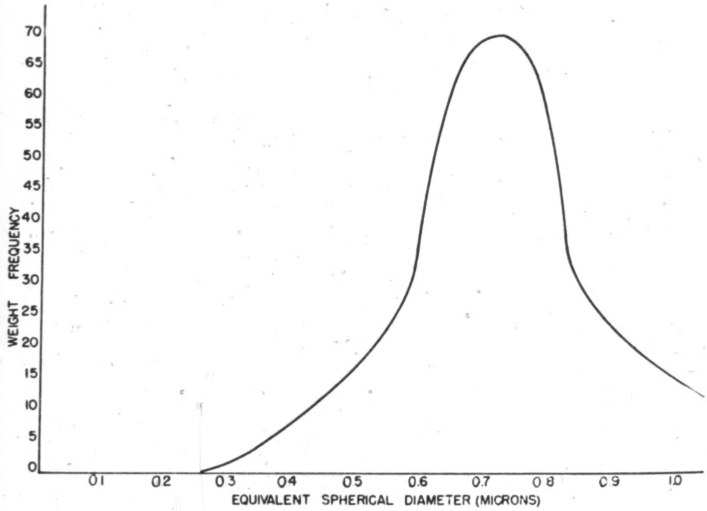

FIG. 9.—Particle-Size Distribution of a Nonsettling Iron Blue Pigment Obtained by Sedimentation of an Aqueous Dispersion in the Sharples Supercentrifuge.

necessary to calculate an equivalent overflow, for example:

$$\text{Equivalent overflow} = \frac{0.194}{10} \times 100 = 1.94$$

Then from Fig. 5 it is found that the length of bowl required for sedimentation of the overflow is 50 cm. The ratios given in the third column of Table III are obtained from a curve such as that in Fig. 4 expanded to $y_{\text{max.}} = 50$ cm.; they are the ratios of the areas under the curve between each centimeter interval to the area under the curve at the 18th centimeter. The fourth column gives the weight of sediment of overflow size obtained from these ratios and the weight of sediment collected in the 18th centimeter. The remaining calculations are like those described above for particle sizes completely sedimented within the bowl.

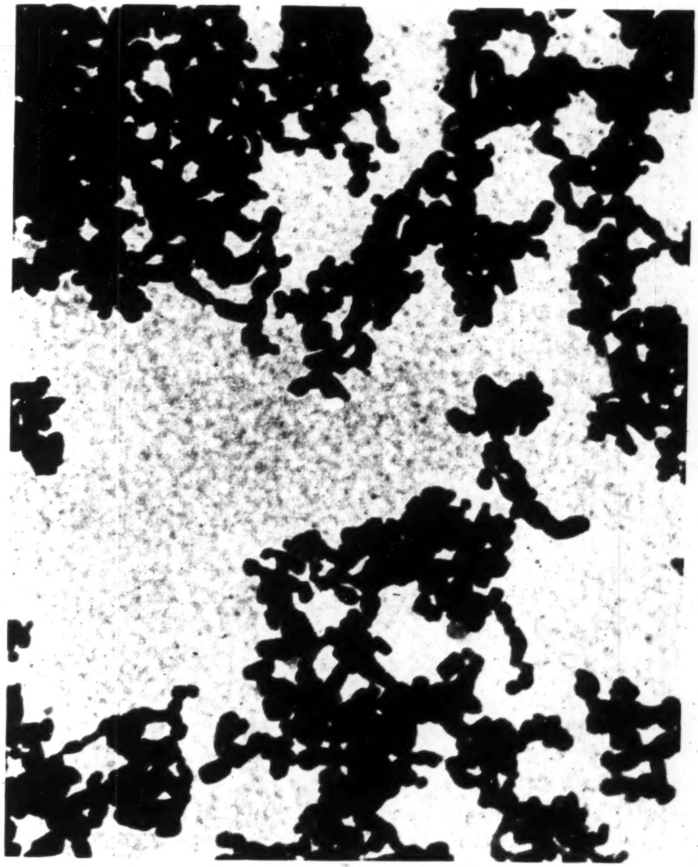

FIG. 10.—Electronmicrograph of a Nonsettling Iron Blue Pigment in an Aqueous Dispersion (× 16,000).

RESULTS

To illustrate the type of results obtained by this method it would, of course, be advantageous to deal with a dispersion of spherical particles of uniform size. The closest approach to these criteria encountered in our work were obtained in dispersion of iron blue pigments in nonaqueous media. The electronmicrograph[4] (Fig. 7) is an example of a dispersion of iron blue in linseed oil. That is, the film for the electronmicrograph was cast from a linseed oil ink of the pigment. While the particle shape is not spherical, it is at least globular; and while the particle size varies, it lies within narrow limits. To obtain comparative results by sedimentation in the Supercentrifuge, the same pigment was ground into linseed oil and diluted with xylene to a pigment concentration of 0.5 per cent. The size distribution obtained by applying the method described above is shown in Fig. 8. The particle sizes indicated in this figure are all found in the electronmicrograph. While we have made no attempt to obtain the size distribution by the electronmicroscopical method, it seems safe to conclude that the particle sizes obtained by the sedimentation method are of the correct order of magnitude.

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[4] Electronmicroscopical work was done by C. Maresh and Ann M. Harding, to whom the thanks of the authors are due.

Another interesting example concerns the determination of the particle-size distribution using a water dispersion a nonsettling iron blue pigment. The surface area of this pigment in the dry state was determined by gas adsorption measurements using the Brunaner-Emmett-Teller (**11**) method of calculation so that some idea of its particle size was available. Actually, the surface area obtained corresponded to that of particles having a mean diameter of 0.06 μ. It was rather perturbing, then, to obtain the size distribution shown in Fig. 9, which indicates the presence of particles about ten times larger. However, the electronmicrograph (Fig. 10) made using a film cast from an aqueous dispersion of this pigment indicates the cause of this apparent discrepancy. The gel-like flocs that are shown in the micrograph apparently sediment as rather stable units. This explains the peculiar settling behavior of iron blue pigments (**12**) from water dispersions and emphasizes the necessity of obtaining good dispersion in order to obtain the correct size distribution of a particular disperse phase.

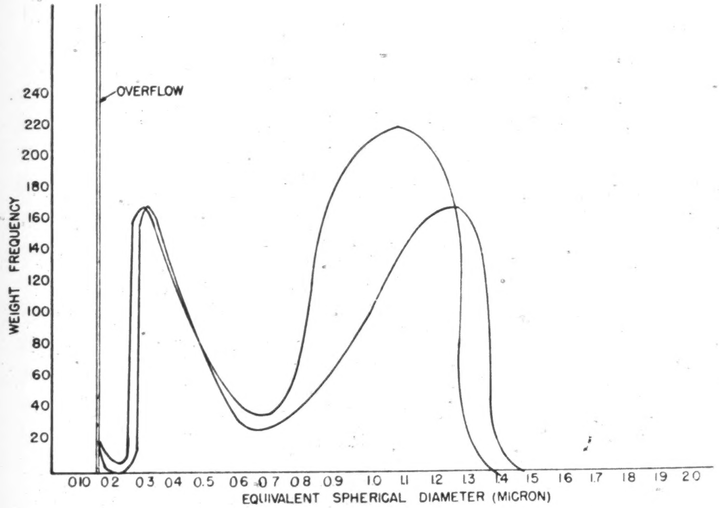

FIG. 11.—Duplicate Determinations of the Particle-Size Distribution of an Ultramarine Pigment in Aqueous Dispersion Obtained by Sedimentation in the Supercentrifuge.

To illustrate the reproducibility of the method, the results of duplicate determinations on a dispersion of an ultramarine pigment in water are shown in Fig. 11. The overflow and finer particle size fraction settled in the bowl agree well in the two experiments. The deviation between the results obtained for the size of the larger particle size fraction is, of course, considerable. It arises chiefly from the fact that this fraction is completely settled in the first few centimeters in the centrifuge bowl, and the sedimentation is greatly influenced by the turbulence in the first 2 cm. of the bowl.

EXPERIMENTAL PROCEDURE

The details of the procedure followed in obtaining the experimental results given above are as follows:

The bowl of the centrifuge used in the sedimentation of aqueous dispersions was lined with a sheet of 0.010-in. vinylite VS 1310 clear; paper was used for nonaqueous systems. A steam-driven No. 1 Sharples centrifuge was used. The centrifuge was brought to the desired speed while pure medium was allowed to flow through the bowl. The speed of the centrifuge was obtained with the 500:1 counting gear supplied with the instrument. The rate of flow was maintained by means of a device based on the Mariotte principle similar to that described by Zentner (**13**). Usually 1 liter of suspension containing 4 to 5 g. of dispersed phase were passed through the centrifuge, the speed and rate of flow being determined near the beginning and again near the end of the passage of the suspension into the bowl. Then a liter of pure medium was allowed to pass through the bowl. In order to prevent redispersion, 1 per cent aluminum sulfate solution was passed through the centrifuge in the case of the aqueous dispersions; ethanol was used for this purpose for the xylene-iron blue dispersions. When the lining was removed from the centrifuge, it was cut into 1-cm. strips using an ordinary paper-cutting board. The sediment was then scraped from these strips into weighing bottles. The weight of the sediment in each centimeter strip was obtained after drying in an oven.

TABLE IV. SPEEDS AND RATES OF FLOW USED TO OBTAIN PARTICLE-SIZE DISTRIBUTIONS.

Dispersion	Speed, rpm.	Rate of flow, cu. cm. per second	Temperature, deg. Cent.
1. Iron Blue—xylene	47 300	2.07	30.0
2. Iron Blue—water	27 300	2.65	21.5
3. Ultramarine—Water (I)	23 500	4.67	24.5
4. Ultramarine—Water (II).	21 900	4.55	25.0

The actual speeds and rates of flow employed in obtaining the four particle-size distributions given above are shown in Table IV.

In calculating the particle sizes, the viscosities of the pure media were used; that of water was obtained from the values in International Critical Tables (**14**) by interpolation. The viscosity of the xylene used was measured with an Ostwald pipet. The densities of the pigments were taken from Gardner (**15**).

CONCLUSION

The method proposed in this paper for calculating the particle-size distribution of a colloidal material from sedimentation data obtained with the Sharples Supercentrifuge is much simpler in its application than the method of Hauser and Reed. It is in better agreement with the type of sedimentation data obtained in the centrifuging of aqueous and nonaqueous pigment dispersions than the modified methods of Hauser and Lynn and Hauser and Schachman. The method seems to give particle sizes of the correct order of magnitude. It gives fairly reproducible results, the reproducibility being better when the experimental conditions can be arranged so that the larger particle sizes are not completely sedimented in the first few centimeters of the bowl. While it may not be possible to recommend it as a precision method, the results obtainable in studies of pigment dispersions such as those illustrated above have been of great value in explaining the behavior of these systems. It is especially valuable when used in conjunction with electron-microscopical and gas adsorption studies as has been done in our work.

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(13) T. Zentner, "Delivery of Liquids at Low and Constant Rates," *Industrial and Engineering Chemistry*, Anal. Ed., Vol. 16, pp. 471–472 (1944).

(14) "International Critical Tables," 1929, McGraw-Hill Book Co., Inc., New York, N. Y., Vol. V, p. 10.

(15) H. A. Gardner, "Physical and Chemical Examination of Paints, Varnishes, Lacquers and Colors," Ninth Edition, Inst. of Paint and Varnish Research, Washington, D. C., p. 283 (1939).

DISCUSSION

Mr. A. R. Lukens.[1]—For some time we have been making particle size distributions on materials down to 0.1μ or less, using a centrifuge method. The paper just presented shows the results of good mathematical approach. I have a suggestion to make that I believe may be helpful. If the computations were reduced to nomograms and then the nomograms used to make a concentric circular slide rule, this might reduce the time required for computation.

Mr. S. J. O'Brien (*author*).—That procedure, of course, was used by Hauser and Schachman. They actually divised such a slide rule for their calculations and it is true that the problem can be greatly simplified by the use of such a device. Also with some of the new calculating machines that are available, the whole method can be made much more accurate.

Mr. Lukens.—What are the man hours required to determine distribution to 0.1μ?

Mr. O'Brien.—I should give it an 8-hr. day for the laboratory and the calculations.

Mr. Lukens.—What means do you use to determine the completeness of dispersion, of a given material in a given vehicle, for the sedimentation?

Mr. O'Brien.—Just trial and error. The example of the sedimentation of iron blue from the aqueous dispersion as given in the paper is an illustration of this difficulty. As in this case the electronmicroscope is often used to help in determining the degree of dispersion obtained.

Mr. Lukens.—Dispersing agents vary in effectiveness. Determination of viscosities against graduated proportions of agents are of interest. Watch to determine the lowest point of the curve. Too much dispersing agent, through building multilayers, may prove confusing.

Mr. O'Brien.—I agree, but we have nothing original to contribute in that line at this time. We have troubles the same as anyone else, making particle size determinations.

Mr. E. W. McMullen.[2]—I should like to ask Mr. O'Brien how he actually makes the weight determination of the material collected in the bowls?

Mr. O'Brien.—The bowl is lined with a plastic of some sort. We use a sheet of vinylite, about 0.01 in. in thickness. After the sedimentation, the liner or sheet is slipped out of the bowl and then it is cut up in the centimeter strips. We use an ordinary paper cutter for this purpose. The sediment, then, can be scraped off the strip of vinylite, put into weighing bottles and weighed. (See also under "Experimental Procedure" in the paper.)

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[1] Vice-President, Powdered Material Research Laboratory, Cambridge, Mass.

[2] Director of Research, The Eagle-Picher Co., Joplin, Mo.

EFFECT OF TEMPERATURE AND HUMIDITY ON MECHANICAL PROPERTIES OF MOLDED CELLULOSE ACETATE PLASTICS*

By W. E. Welch,[1] R. F. Hayes[1,2] T. S. Carswell,[1,3] and H. K. Nason[1,4]

Synopsis

Four molding compound formulations, covering a range of flow gradings, were prepared from medium-acetyl (37.7 per cent) and four from high-acetyl (41.0 per cent) cellulose acetate. Test specimens were injection-molded from these formulations and physical properties were determined by standard A.S.T.M. procedures. The effects on yield and ultimate strengths in tension, elongation, modulus of elasticity, yield strength in flexure, and impact strength, resulting from variations in ambient temperature over the range from −25 C. (−13 F.) to 80 C. (176 F.), and of variation in relative humidity at 25 C. (77 F.) over the range from zero per cent to saturation, were determined and the results are shown in graphical form. Some data showing the effect of exposure to weather on tensile properties of two of the formulations are presented also. The practical importance of these data is discussed.

In recent years, plastic materials have been used to an increasing extent for applications where dependable performance must be assured over a wide range of ambient atmospheric conditions. Intelligent engineering design for such applications requires data on the mechanical behavior of the materials under all conditions which may be encountered in service, and such data have been scarce. The work reported herein was undertaken to provide information on the mechanical properties of cellulose acetate molding compositions over the customary range of atmospheric temperatures and humidities.

Materials

Eight typical cellulose acetate molding compositions were prepared by standard commercial techniques—four from cellulose acetate of medium acetyl content (37.7 per cent) and four from cellulose acetate of high acetyl content (41.0 per cent). Mixtures of phthalate and phosphate esters were used as plasticizers. The finished compositions, which were representative of commercial products, are further characterized in Table I.

Specimens were injection-molded on a 2-oz. Reed Prentice machine, using predried compositions (1.5 to 2 hr. at 200 F.) under conditions experimentally determined to be optimum for each material and molded shape. The moldings were thoroughly randomized before selecting groups of specimens for the various tests.

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[*] Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

[1] Research Dept., Monsanto Chemical Co., Plastics Div., Springfield, Mass.

[2] Present address: Gering Products Co., Kenilworth, N. J.

[3] Present address: Manager, Research and Development Dept., Commercial Solvents Corp., Terre Haute, Ind.

[4] Present address: Monsanto Chemical Co., Central Research Dept., Dayton, Ohio.

TEST METHODS

A.S.T.M. test methods were used throughout. The pertinent references are given below:

Tensile Properties.—A.S.T.M. Tentative Method of Test for Tensile Properties of Plastics (D 638 – 41 T)[5], type II specimen. Tests were run at a crosshead speed of 0.2 in. per minute, using a Tinius Olsen Universal testing machine. Data were obtained on yield strength, ultimate strength, and ultimate elongation; and stress-strain curves, from which moduli of elasticity were calculated, were obtained also. At least six tests were made at every environmental condition.

TABLE I. CELLULOSE ACETATE MOLDING COMPOSITIONS.

Sample No.	A.S.T.M. Classification[a]		A.S.T.M. Flow Temperature		Acetyl Content of Base Acetate, per cent	Plasticizer Content, per cent[b]	A.S.T.M. Distortion Temperature	
	Type	Grade	deg. Cent.	deg. Fahr.			deg. Cent.	deg. Fahr.
No. 1	II	7	158	316	37.7	28.6	75.5	168
No. 4	I	2	149	300	37.7	28.4	73.0	163
No. 2	III	9	146	295	37.7	32.3	72.5	163
No. 3	III	10	135	275	37.7	37.2	66.0	151
No. 5	IV	13	160	320	41.0	25.0	69.5	157
No. 8	IV	14	152	306	41.0	27.0	67.5	154
No. 6	IV	15	145	293	41.0	30.9	60.0	145
No. 7	III	10	134	273	41.0	35.6	50.5	123

[a] A.S.T.M. Tentative Specifications for Cellulose Acetate Molding Compounds (D 706 – 44 T), 1944 Book of A.S.T.M. Standards, Part III, p. 1549. Conformance to these classifications is not exact in every case, but the materials have been placed in the group to which they most nearly belong.

[b] Samples Nos. 1, 2, 3, 5, 6, and 7 contain a mixture of phthalate and phosphate esters; samples Nos. 4 and 8 contain phthalate esters only.

Flexural Properties.—A.S.T.M. Tentative Method of Test for Flexural Strength of Electrical Insulating Materials (D 650 – 42 T).[6] Specimens 0.5 by 0.5 by 5.0 in. were used. The flexure jig was adjusted to give a span-depth ratio of 8:1, and tests were made at a crosshead speed of 0.05 in. per minute. Maximum fiber stress was calculated by the usual formula. At least five tests were made at each condition.

Impact Strength.—A.S.T.M. Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (D 256 – 43 T).[7] Specimens 0.5 by 0.5 by 5.0 in., with a machined notch in the center, were broken by the Charpy method, using a Tinius Olsen impact tester of 100 in-lb. capacity. Appropriate notches were then machined into the broken halves, and these, after reconditioning, were tested by the Izod method using a Bell Laboratories (Baldwin-Southwark) impact tester. All notching was done at 400 rpm. with a single-tooth diamond milling cutter, and representative specimens were examined microscopically to insure conformance with specifications for contour of the notch. At least five Charpy tests and ten Izod tests were made at each condition.

Distortion Temperature.—A.S.T.M. Tentative Method of Test for Heat Distortion Temperature of Plastics (D 648 – 41 T).[8] Specimens 0.5 by 0.5 by 5.0 in. were conditioned at 50 C. (122 F.) for 48 hr., cooled to room temperature in a desiccator over calcium chloride and stored therein until tested.

Flow Temperature.—A.S.T.M. Tentative Method of Test for Measuring the Flow Properties of Thermoplastic Molding Materials (D 569 – 44 T).[9] Molding composition granules were conditioned for 25 min. at 100 C. (212 F.), cooled in a desiccator over calcium chloride, formed into pellets $\frac{3}{8}$ in. in diameter and $\frac{3}{8}$ in. high in a pelleting press, and tested in the Rossi-Peakes apparatus at 1500 psi. Determinations were made at several temperatures, the results were plotted and the flow temperatures determined by interpolation as prescribed.

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[5] 1944 Book of A.S.T.M. Standards, Part III, p. 1682.

[6] *Ibid*, p. 1399.

[7] *Ibid.*, p. 1402.

[8] *Ibid.*, p. 1627.

[9] *Ibid.*, p. 1647.

Moisture Content.—Actual water content of conditioned specimens was determined by planing into thin chips, soaking these in methanol, and determining water by titration with Karl Fischer reagent (**1**).[10] A blank was run with methanol alone and the value obtained subtracted from all determinations.

CONDITIONING

All specimens were taken directly from the molding machine, placed in sealed metal containers over anhydrous calcium chloride and allowed to remain until needed. Specimens for flow temperature and distortion temperature tests were conditioned as described in the preceding section.

For the mechanical tests at various temperatures, the dry specimens were heated in a circulating air oven or cooled in a special chamber using dry ice as the refrigerant, as required, until thermal equilibrium was established but in no case for less than 1 hr. They were then transferred quickly to the testing machine. Impact tests were run immediately upon completion of the transfer. For tension and flexure tests, temperature-controlled enclosures which have been described in previous publications (**2**, **3**) were used, and specimens were stored within these enclosures until needed for testing.[11] In every case, the test specimens were within ±3 C. (±5.4 F.) of the nominal temperatures during testing.

For the mechanical tests at various relative humidities, specimens were stored at the desired humidity at 25 C. (77 F.) for 3 to 7 weeks, depending upon thickness. Representative samples at each condition were taken for analysis at the time of testing. Relative humidity conditions of 20 and 35 per cent were obtained in a special test room, the air of which was conditioned by mechanical means. Relative humidity of 50 per cent was obtained in the main physical laboratory, whose atmosphere is maintained at the standard laboratory conditions of 25 ± 1.1 C. (77 ± 2 F.) and 50 ± 2 per cent relative humidity in accordance with A.S.T.M. Tentative Methods of Conditioning and Classifying for Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 44 T).[12] Other conditions were obtained by storage of the specimens in sealed containers over appropriate aqueous solutions: saturated sodium nitrate for 65 per cent, saturated ammonium chloride for 80 per cent, and distilled water for a saturated atmosphere (sometimes erroneously designated as "100 per cent relative humidity"). The relative humidity for each condition was checked with a standardized hair hygrometer and is believed to have been within ±2 per cent of the nominal value in every case. Specimens were tested as rapidly as possible after removal from the conditioning chamber.

Despite the long time allowed for conditioning, it is obvious from comparison of moisture contents that the thicker specimens had not reached true equilibrium.

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[10] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1464.

[11] Tentative Methods of Test for Tensile and Compressive Properties of Plastics at Subnormal and Supernormal Temperatures, 1944 Book of A.S.T.M. Standards, Part III, p. 1693.

[12] 1944 Book of A.S.T.M. Standards, Part III, p. 1385.

TABLE II.—EFFECT OF TEMPERATURE ON TENSILE PROPERTIES; INJECTION-MOLDED CELLULOSE ACETATE.

Formulation (Table I)		No. 1	No. 4	No. 2	No. 3	No. 5	No. 8	No. 6	No. 7
A.S.T.M. Type[a]		II, 7	I, 2	III, 9	III, 10	IV, 13	IV, 14	IV, 15	III, 10
−25 C. (−13 F.)	Moisture content, per cent	0.17	0.21	0.17	0.18	0.20	0.17	0.21	0.16
	Tensile yield stress, psi.	...	...	...	...	...	...	...	...
	Ultimate tensile stress, psi.	8540	9400	7950	6490	8710	9380	7710	6370
	Elongation at break, per cent	3.2	3.9	3.0	4.4	2.8	4.5	4.6	3.6
0 C. (32 F.)	Moisture content, per cent	0.18	0.16	0.15	0.17	0.16	0.19	0.13	0.12
	Tensile yield stress, psi.	9570	8200	7530	5690	9190	7790	7040	5670
	Ultimate tensile stress, psi.	9300	8060	7410	5730	9030	...	6820	5490
	Elongation at break, per cent	13.3	12.8	16.4	18.3	5.9	12.5	17.4	12.0
25 C. (77 F.)	Moisture content, per cent	0.18	0.19	0.14	0.13	0.12	0.13	0.11	0.09
	Tensile yield stress, psi.	6250	5640	4750	3530	6150	5300	4370	3510
	Ultimate tensile stress, psi.	5880	5100	4860	3430	5510	4600	4440	3190
	Elongation at break, per cent	22.9	23.9	27.9	38.5	15.6	19.8	42.6	38.1
50 C. (122 F.)	Moisture content, per cent	0.33	0.38	0.36	0.21	0.29	0.37	0.38	0.29
	Tensile yield stress, psi.	3660	3390	2690	2040	3480	3080	2405	1710
	Ultimate tensile stress, psi.	3730	3080	2570	2160	3250	2514	2500	1620
	Elongation at break, per cent	30.3	27.1	31.6	43.0	32.0	22.6	37.7	32.9
65 C. (149 F.)	Moisture content, per cent	0.08	0.11	0.10	0.12	0.07	0.09	0.11	0.11
	Tensile yield stress, psi.	2900	2590	1860	1140	2680	2150	1380	830
	Ultimate tensile stress, psi	2540	2160	1610	1200	2150	1610	1370	850
	Elongation at break, per cent	29.8	25.4	30.9	41.5	25.8	17.1	33.1	31.2
80 C. (176 F.)	Moisture content, per cent	0.16	0.30	0.15	0.28	0.20	0.19	0.18	0.17
	Tensile yield stress, psi.	2140	2110	1390	550	1780	1530	490	210
	Ultimate tensile stress, psi.	2140	2110	1390	550	1780	1530	490	210
	Elongation at break, per cent	21.2	11.1	22.7	33.7	15.8	14.6	33.4	31.4

[a] Tentative Specifications for Cellulose Acetate Molding Compounds (D 706 – 44 T), 1944 Book of A.S.T.M. Standards, Part III, p. 1549.

TABLE III.—EFFECT OF RELATIVE HUMIDITY AT 25 C. (77 F.) ON TENSILE PROPERTIES; INJECTION-MOLDED CELLULOSE ACETATE.

Formulation (Table I)		No. 1	No. 4	No. 2	No. 3	No. 5	No. 8	No. 6	No. 7
A.S.T.M. Type[a]		II, 7	I, 2	III, 9	III, 10	IV, 13	IV, 14	IV, 15	III, 10
Dry (0 per cent relative humidity)	Moisture content of specimen, per cent	0.18	0.19	0.14	0.13	0.12	0.13	0.11	0.09
	Tensile yield stress, psi.	6250	5640	4750	3530	6150	5300	4370	3510
	Ultimate tensile stress, psi.	5880	5100	4860	3430	5510	4600	4440	3190
	Elongation at break, per cent	22.9	23.9	27.9	38.5	15.6	19.8	42.6	38.1
20 per cent relative humidity	Moisture content of specimen, per cent	0.55	0.55	0.44	0.39	0.43	0.41	0.34	0.32
	Tensile yield stress, psi.	5770	5010	4450	3400	5740	4910	4140	3380
	Ultimate tensile stress, psi.	5230	4360	3870	3160	5060	4170	4000	2910
	Elongation at break, per cent	23.2	21.9	26.9	38.0	18.5	17.4	38.9	34.0
35 per cent relative humidity	Moisture content of specimen, per cent	0.82	0.81	0.72	0.63	0.64	0.57	0.51	0.45
	Tensile yield stress, psi.	5350	4900	4270	3300	5310	4740	3990	3340
	Ultimate tensile stress, psi.	4720	4220	3730	3070	4440	3900	3780	2890
	Elongation at break, per cent	25.9	20.8	24.7	36.8	28.4	18.9	41.8	37.6
50 per cent relative humidity	Moisture content of specimen, per cent	1.36	1.45	1.19	1.12	1.08	1.07	0.95	0.88
	Tensile yield stress, psi.	4270	4000	3510	2540	4530	4050	3300	2740
	Ultimate tensile stress, psi.	4270	4000	3100	2380	4530	3370	3300	2740
	Elongation at break, per cent	31.6	26.2	27.9	43.7	30.3	22.3	44.0	39.9
65 per cent relative humidity	Moisture content of specimen, per cent	1.92	1.90	1.71	1.54	1.47	1.48	1.31	1.22
	Tensile yield stress, psi.	4080	3860	3330	2510	4390	3910	3240	2510
	Ultimate tensile stress, psi.	3460	3040	2620	2160	3580	3110	3040	2060
	Elongation at break, per cent	30.3	23.3	31.4	46.4	28.6	18.0	45.9	37.9
90 per cent relative humidity	Moisture content of specimen, per cent	2.65	2.62	2.44	2.15	2.08	1.99	1.81	1.72
	Tensile yield stress, psi.	3380	3200	2730	2020	3800	3410	2730	2200
	Ultimate tensile stress, psi.	2890	2520	2200	1830	3080	2660	2700	1900
	Elongation at break, per cent	35.5	28.1	38.3	52.2	31.5	16.7	49.5	42.9
Saturated atmosphere	Moisture content of specimen, per cent	5.50	5.56	5.06	4.68	3.67	3.84	3.32	3.12
	Tensile yield stress, psi.	2320	1980	1820	1410	2930	2610	2050	1410
	Ultimate tensile stress, psi.	1480	1600	1390	1070	2230	2000	2040	1380
	Elongation at break, per cent	37.8	22.4	32.8	27.5	31.2	14.3	55.7	52.7

[a] Tentative Specifications for Cellulose Acetate Molding Compounds (D 706 – 44 T), 1944 Book of A.S.T.M. Standards, Part III, p. 1549.

TEST RESULTS

Typical stress-strain curves (in this case for formulation No. 1) at the various test temperatures are shown in Fig. 1. The initial portions of these curves are shown to a larger scale in Fig. 2.

Similarly, typical stress-strain curves for this same formulation at various relative humidities at 25 C. (77 F.) are shown in Fig. 3.

The data on the tensile properties of "dry" specimens at various temperatures are summarized in Table II and the corresponding data at various relative humidities are summarized in Table III. Moisture content at the time of testing is shown for each formulation and at each

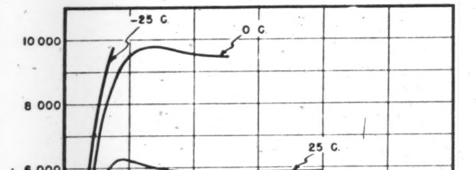

FIG. 1.—Typical Stress-Strain Curves for Injection-Molded Cellulose Acetate. (Formulation No. 1; A.S.T.M. Type II, Grade 7.)

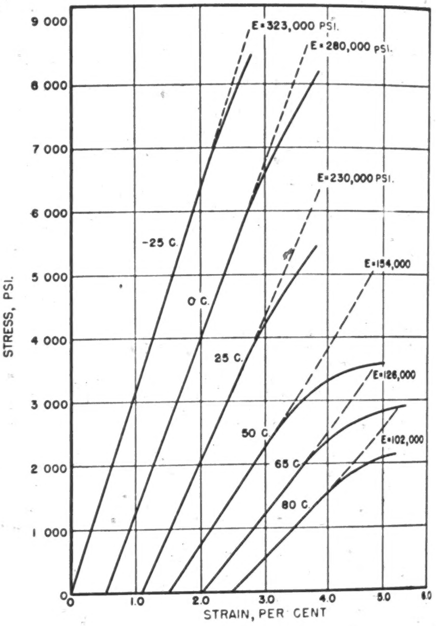

FIG. 2.—Typical Initial Portion of Stress Strain Curves for Injection-Molded Cellulose Acetate. (Formulation No. 1; A.S.T.M. Type II, Grade 7.)

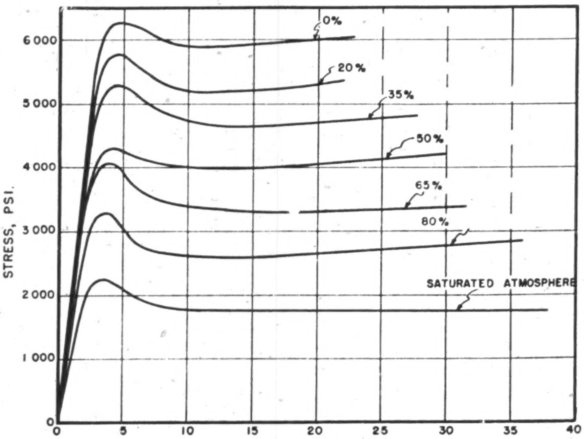

FIG. 3.—Effect of Humidity on Stress-Strain Curve for Injection-Molded Cellulose Acetate. (Formulation No. 1; A.S.T.M. Type II, Grade 7.)

test condition. Stress values shown are nominal values in every case.[13] The corresponding data on moduli of elasticity are summarized in Tables IV and V.

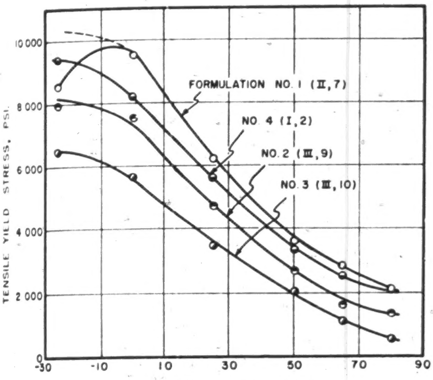

Fig. 4.—Effect of Temperature on Tensile Yield Stress of Injection-Molded Cellulose Acetate. (Medium-Acetyl Formulations.)

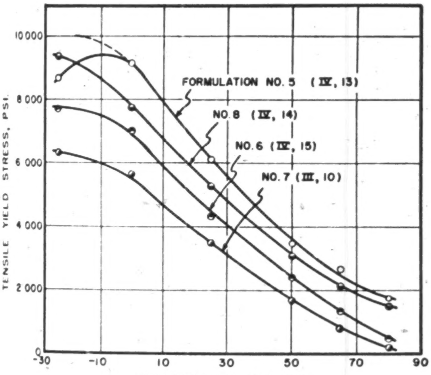

Fig. 5.—Effect of Temperature on Tensile Yield Stress of Injection-Molded Cellulose Acetate. (High-Acetyl Formulations.)

Figures 4 and 5 show in graphical form the influence of temperature on the upper yield stress of these materials, and Figs. 6 and 7 summarize similar information on ultimate elongation. In Fig. 8, the effect of temperature on modulus of elasticity is shown for the medium-acetyl formulations. Data for the high-acetyl formulations are not shown since they follow the same trends at generally lower levels.

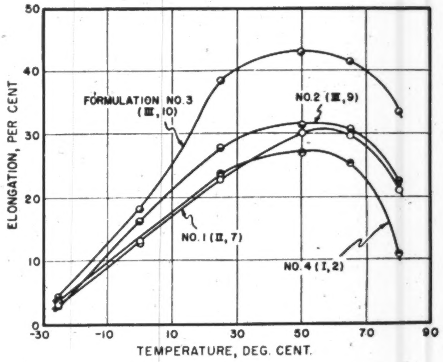

Fig. 6.—Effect of Temperature on Elongation of Injection-Molded Cellulose Acetate. (Medium-Acetyl Formulations.)

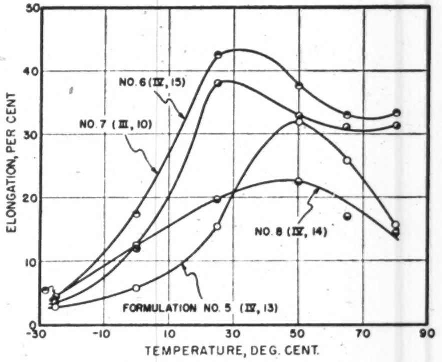

Fig. 7.—Effect of Temperature on Elongation of Injection-Molded Cellulose Acetate (High-Acetyl Formulations.)

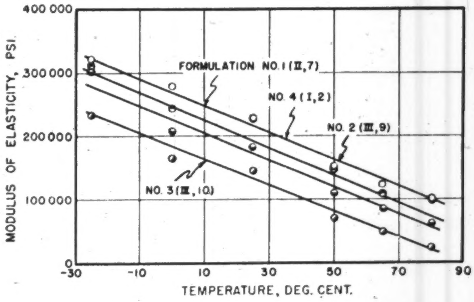

Fig. 8.—Effect of Temperature on Modulus of Elasticity of Injection-Molded Cellulose Acetate. (Medium-Acetyl Formulations.)

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[13] A.S.T.M. Method D 638 – 44 T (see footnote 5) describes the terms used in this paper.

TABLE IV.—EFFECT OF TEMPERATURE ON MODULUS OF ELASTICITY; INJECTION-MOLDED CELLULOSE ACETATE.

Formulation (Table I)		No. 1	No. 4	No. 2	No. 3	No. 5	No. 8	No. 6	No. 7
A.S.T.M. Type[a]		II, 7	I, 2	III, 9	III, 10	IV, 13	IV, 14	IV, 15	III, 10
−25 C. (−13 F.)	Water, per cent	0.17	0.21	0.17	0.18	0.20	0.17	0.21	0.16
	$E \times 10^{-5}$	3.23	3.13	3.03	2.33	3.27	2.96	2.06	2.50
0 C. (32 F.)	Water, per cent	0.18	0.16	0.15	0.17	0.16	0.19	0.13	0.12
	$E \times 10^{-5}$	2.80	2.44	2.09	1.69	2.77	2.34	2.02	1.93
25 C. (77 F.)	Water, per cent	0.18	0.19	0.14	0.13	0.12	0.13	0.11	0.09
	$E \times 10^{-5}$	2.30	2.28	1.84	1.47	2.37	2.18	1.70	1.45
50 C. (122 F.)	Water, per cent	0.33	0.38	0.36	0.21	0.29	0.37	0.38	0.29
	$E \times 10^{-5}$	1.54	1.49	1.11	0.71	1.41	1.36	0.92	0.60
65 C. (149 F.)	Water, per cent	0.08	0.11	0.10	0.12	0.07	0.09	0.11	0.11
	$E \times 10^{-5}$	1.26	1.10	0.87	0.50	1.24	1.08	0.59	0.38
80 C. (176 F.)	Water, per cent	0.16	0.30	0.15	0.28	0.20	0.19	0.18	0.17
	$E \times 10^{-5}$	1.02	1.00	0.63	0.25	0.78	0.65	0.20	0.10

[a] Tentative Specifications for Cellulose Acetate Molding Compounds (D 706 - 44 T), 1944 Book of A.S.T.M. Standards, Part III, p. 1549.

TABLE V.—EFFECT OF RELATIVE HUMIDITY AT 25 C. (77 F.) ON MODULUS OF ELASTICITY; INJECTION-MOLDED CELLULOSE ACETATE.

Formulation (Table I)		No. 1	No. 4	No. 2	No. 3	No. 5	No. 8	No. 6	No. 7
A.S.T.M. Type[a]		II, 7	I, 2	III, 9	III, 10	IV, 13	IV, 14	IV, 15	III, 10
Dry (0 per cent relative humidity)	Water, per cent	0.18	0.19	0.14	0.13	0.12	0.13	0.11	0.09
	$E \times 10^{-5}$	2.30	2.28	1.84	1.47	2.37	2.18	1.70	1.45
20 per cent relative humidity	Water, per cent	0.55	0.55	0.44	0.39	0.43	0.41	0.34	0.32
	$E \times 10^{-5}$	2.12	2.05	1.76	1.28	2.11	1.96	1.61	1.37
35 per cent relative humidity	Water, per cent	0.82	0.81	0.72	0.63	0.64	0.57	0.51	0.45
	$E \times 10^{-5}$	1.99	1.86	1.67	1.27	1.77	1.84	1.56	1.35
50 per cent relative humidity	Water, per cent	1.36	1.45	1.19	1.12	1.08	1.07	0.95	0.88
	$E \times 10^{-5}$	1.72	1.69	1.46	1.17	1.69	1.65	1.39	1.10
65 per cent relative humidity	Water, per cent	1.92	1.90	1.71	1.54	1.47	1.48	1.31	1.22
	$E \times 10^{-5}$	1.61	1.61	1.29	1.03	1.61	1.60	1.25	1.02
80 per cent relative humidity	Water, per cent	2.65	2.62	2.44	2.15	2.08	1.99	1.81	1.72
	$E \times 10^{-5}$	1.47	1.41	1.23	0.89	1.58	1.47	1.24	0.93
Saturated atmosphere	Water, per cent	5.50	5.56	5.06	4.68	3.67	3.84	3.32	3.12
	$E \times 10^{-5}$	0.99	1.02	0.83	0.62	1.36	1.23	0.95	0.68

[a] Tentative Specifications for Cellulose Acetate Molding Compounds (D 706 - 44 T), 1944 Book of A.S.T.M. Standards, Part III, p. 1549.

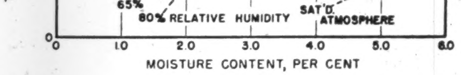

FIG. 9.—Effect of Relative Humidity on Tensile Yield Stress of Injection-Molded Cellulose Acetate. (Medium-Acetyl Formulations.)

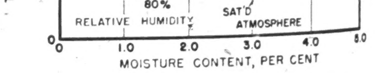

FIG. 10.—Effect of Relative Humidity on Tensile Yield Stress of Injection-Molded Cellulose Acetate. (High-Acetyl Formulations.)

The effect of humidity on upper yield stress is shown in Figs. 9 and 10, on ultimate elongation in Figs. 11 and 12, and on modulus of elasticity for the medium

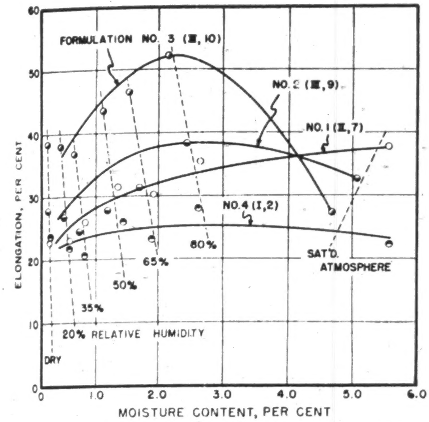

Fig. 11.—Effect of Relative Humidity on Elongation of Injection-Molded Cellulose Acetate. (Medium-Acetyl Formulations.)

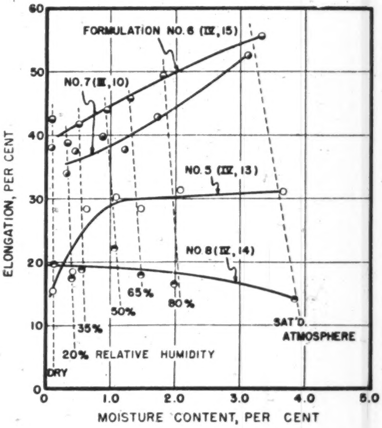

Fig. 12.—Effect of Relative Humidity on Elongation of Injection-Molded Cellulose Acetate. (High-Acetyl Formulations.)

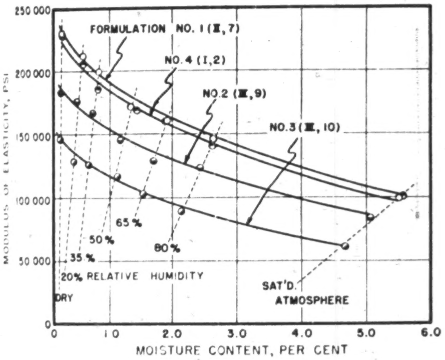

Fig. 13.—Effect of Relative Humidity on Modulus of Elasticity of Injection-Molded Cellulose Acetate. (Medium-Acetyl Formulations.)

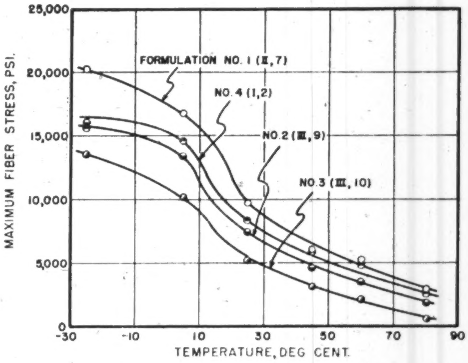

Fig. 14.—Effect of Temperature on Flexural Strength of Injection-Molded Cellulose Acetate. (Medium-Acetyl Formulations.)

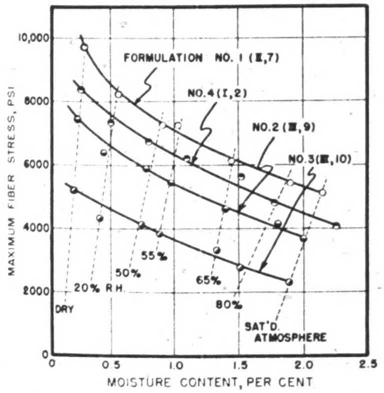

Fig. 15.—Effect of Relative Humidity on Flexural Strength of Injection-Molded Cellulose Acetate. (Medium-Acetyl Formulations.)

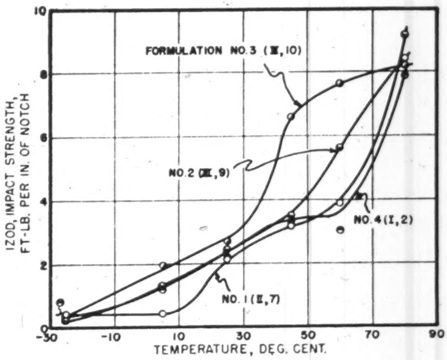

Fig. 16.—Effect of Temperature on Impact Strength of Injection-Molded Cellulose Acetate. (Medium-Acetyl Formulations.)

acetyl formulations in Fig. 13. (The curves for the high-acetyl formulas are very similar.) The abscissas represent moisture contents of the specimens when tested; relative humidity contours are shown by the broken lines.

TABLE VI.—EFFECT OF TEMPERATURE ON FLEXURAL STRENGTH; INJECTION-MOLDED CELLULOSE ACETATE.

Formulation (Table I)		No. 1	No. 4	No. 2	No. 3	No. 5	No. 8	No. 6	No. 7
A.S.T.M. Type[a]		II, 7	I, 2	III, 9	III, 10	IV, 13	IV, 14	IV, 15	III, 10
−25 C. (−13 F.)	Water content, per cent	0.11	0.32	0.25	0.26	0.36	0.40	0.32	0.27
	Maximum fiber stress, psi	20 240	15 580	16 120	13 520	16 310	16 590	15 340	12 870
5 C. (41 F.)	Water content, per cent	...	...	...	...	...	...	...	...
	Maximum fiber stress, psi	16 720	14 640	13 440	10 180	8 770	12 890	11 700	10 020
25 C. (77 F.)	Water content, per cent	0.29	0.27	0.23	0.20	0.23	0.19	0.21	0.18
	Maximum fiber stress, psi	9720	8380	7420	5210	8890	8090	6860	5140
45 C. (113 F.)	Water content, per cent	0.23	0.22	0.18	0.23	0.19	0.19	...	0.17
	Maximum fiber stress, psi	6080	5840	4650	3120	6040	5210	4280	2710
60 C. (140 F.)	Water content, per cent	0.16	0.13	0.27	0.12	0.12	0.18	0.13	0.11
	Maximum fiber stress, psi	5260	4810	3520	2100	5880	4270	2940	1460
80 C. (176 F.)	Water content, per cent	...	...	...	...	...	...	...	...
	Maximum fiber stress, psi	2950	2500	1790	510	2480	1050	1150	0

[a] Tentative Specifications for Cellulose Acetate Molding Compounds (D 706 – 44 T), 1944 Book of A.S.T.M. Standards, Part III, p. 1549.

TABLE VII.—EFFECT OF RELATIVE HUMIDITY AT 25 C. (77 F.) ON FLEXURAL STRENGTH; INJECTION-MOLDED CELLULOSE ACETATE.

Formulation (Table I)		No. 1	No. 4	No. 2	No. 3	No. 5	No. 8	No. 6	No. 7
A.S.T.M. Type[a]		II, 7	I, 2	III, 9	III, 10	IV, 13	IV, 14	IV, 15	III, 10
Dry (0 per cent relative humidity)	Water content, per cent	0.29	0.27	0.23	0.20	0.23	0.19	0.21	0.18
	Maximum fiber stress, psi	9720	8380	7420	5210	8890	8090	6860	5140
20 per cent relative humidity	Water content, per cent	0.57	0.51	0.45	0.42	0.49	0.42	0.40	0.36
	Maximum fiber stress, psi	8220	7330	6400	4330	8200	7010	6080	4410
50 per cent relative humidity	Water content, per cent	0.92	0.81	0.78	0.75	0.70	0.74	0.62	0.63
	Maximum fiber stress, psi	7270	6770	5900	4160	7780	6770	5890	4220
55 per cent relative humidity	Water content, per cent	1.03	1.11	0.98	0.89	0.84	0.86	0.79	0.65
	Maximum fiber stress, psi	7290	6220	5440	3840	7540	6800	5720	4030
65 per cent relative humidity	Water content, per cent	1.45	1.52	1.41	1.34	1.25	1.21	1.15	1.10
	Maximum fiber stress, psi	6140	5630	4620	3370	6790	5740	5010	3450
80 per cent relative humidity	Water content, per cent	1.91	1.78	1.82	1.51	1.59	1.64	1.47	1.35
	Maximum fiber stress, psi	5460	4810	4170	2790	6210	5220	4110	2970
Saturated atmosphere	Water content, per cent	2.15	2.26	2.00	1.89	1.81	1.74	1.55	1.89
	Maximum fiber stress, psi	5120	4070	3680	2330	5880	4680	3920	2640

[a] Tentative Specifications for Cellulose Acetate Molding Compounds (D 706 – 44 T), 1944 Book of A.S.T.M. Standards, Part III, p. 1549.

The effects of temperature and of relative humidity on flexural strength are shown in Tables VI and VII. These effects are shown graphically in Figs. 14 and 15 for the medium-acetyl formulations.

Data on the effect of temperature on impact strength are summarized in Table VIII, and the Izod data are shown graphically in Fig. 16 for the medium acetyl formulations. The corresponding data on the effect of humidity are summarized in Table IX, and the Izod values

TABLE VIII.—EFFECT OF TEMPERATURE ON IMPACT STRENGTH; INJECTION-MOLDED CELLULOSE ACETATE.

Formulation (Table I)		No. 1	No. 4	No. 2	No. 3	No. 5	No. 8	No. 6	No. 7
A.S.T.M. Type[a]		II, 7	I, 2	III, 9	III, 10	IV, 13	IV, 14	IV, 15	III, 10
−25 C. (−13 F.)	Water content, per cent	0.11	0.32	0.25	0.26	0.36	0.40	0.32	0.27
	Izod, ft-lb. per inch of notch	0.42	0.42	0.27	0.31	0.34	0.41	0.47	0.37
	Charpy, ft-lb. per inch of notch	0.47	0.39	0.34	0.37	0.33	0.36	0.51	0.41
5 C. (41 F.)	Water content, per cent	...	...	...	...	...	...	...	...
	Izod, ft-lb. per inch of notch	0.45	1.20	1.32	1.96	0.52	1.24	1.50	1.72
	Charpy, ft.-lb per inch of notch	0.49	1.28	1.20	2.27	0.51	1.80	1.63	2.15
25 C. (77 F.)	Water content, per cent	0.29	0.27	0.23	0.20	0.23	0.19	0.21	0.18
	Izod, ft-lb. per inch of notch	2.14	2.17	2.42	2.71	1.82	1.89	2.06	2.48
	Charpy, ft-lb. per inch of notch	2.64	2.94	3.13	3.39	2.35	2.85	2.90	3.21
45 C. (113 F.)	Water content, per cent	0.23	0.22	0.18	0.23	0.19	0.19	...	0.17
	Izod, ft-lb. per inch of notch	3.18	3.32	3.51	6.61	3.43	3.06	4.14	7.18
	Charpy, ft-lb. per inch of notch	4.51	4.40	4.48	5.37	3.87	4.48	5.04	5.65
60 C. (140 F.)	Water content, per cent	0.16	0.13	0.27	0.12	0.12	0.18	0.13	0.11
	Izod, ft-lb. per inch of notch	3.87	3.05	5.63	7.64	3.44	3.46	7.54	7.65
	Charpy, ft-lb. per inch of notch	5.10	3.82	4.37	6.28	4.10	4.36	5.56	7.21
80 C. (176 F.)	Water content, per cent	...	...	...	...	...	...	...	...
	Izod, ft-lb. per inch of notch	9.17	7.90	8.41	8.20	8.45	9.05	8.83	7.86
	Charpy, ft-lb. per inch of notch	9.10	5.08	7.27	7.49	7.09	8.93	9.09	7.16

[a] Tentative Specifications for Cellulose Acetate Molding Compounds (D 706 – 44 T), 1944 Book of A.S.T.M. Standards, Part III, p. 1549.

TABLE IX.—EFFECT OF RELATIVE HUMIDITY AT 25 C. (77 F.) ON IMPACT STRENGTH; INJECTION-MOLDED CELLULOSE ACETATE.

Formulation (Table I)		No. 1	No. 4	No. 2	No. 3	No. 5	No. 8	No. 6	No. 7
A.S.T.M. Type[a]		II, 7	I, 2	III, 9	III, 10	IV, 13	IV, 14	IV, 15	III, 10
Dry (0 per cent relative humidity)	Water content, per cent	0.29	0.27	0.23	0.20	0.23	0.19	0.21	0.18
	Izod, ft-lb. per inch of notch	2.14	2.17	2.42	2.71	1.82	1.89	2.09	2.48
	Charpy, ft-lb. per inch of notch	2.64	2.94	3.13	3.39	2.35	2.85	2.90	3.21
20 per cent relative humidity	Water content, per cent	0.57	0.51	0.45	0.42	0.49	0.42	0.40	0.36
	Izod, ft-lb. per inch of notch	2.62	2.60	2.61	3.15	2.03	2.31	2.62	2.59
	Charpy, ft-lb. per inch of notch	3.43	3.28	3.54	3.98	2.92	3.33	3.68	3.73
50 per cent relative humidity	Water content, per cent	0.92	0.81	0.78	0.75	0.70	0.73	0.62	0.63
	Izod, ft-lb. per inch of notch	2.70	2.38	2.74	3.14	2.08	2.45	2.82	2.98
	Charpy, ft-lb. per inch of notch	3.75	3.27	3.61	3.97	3.09	3.40	3.86	3.61
55 per cent relative humidity	Water content, per cent	1.03	1.11	0.98	0.89	0.84	0.86	0.79	0.65
	Izod, ft-lb. per inch of notch	3.19	2.88	3.01	3.46	2.45	2.39	3.25	3.14
	Charpy, ft-lb. per inch of notch	4.51	3.59	4.08	4.08	3.28	3.25	4.28	4.08
65 per cent relative humidity	Water content, per cent	1.45	1.52	1.41	1.35	1.25	1.21	1.15	1.10
	Izod, ft-lb. per inch of notch	3.43	2.95	3.34	6.32	2.73	2.92	3.59	3.41
	Charpy, ft-lb. per inch of notch	4.48	3.85	4.18	5.05	3.70	3.97	4.70	3.94
80 per cent relative humidity	Water content, per cent	1.91	1.78	1.82	1.51	1.59	1.64	1.47	1.35
	Izod, ft-lb. per inch of notch	3.91	3.67	4.00	6.76	3.09	3.26	3.92	4.38
	Charpy, ft-lb. per inch of notch	4.93	4.58	4.64	6.26	3.79	3.93	4.79	4.27
100 per cent relative humidity	Water content, per cent	2.15	2.26	2.00	1.89	1.81	1.89	1.74	1.55
	Izod, ft-lb. per inch of notch	4.40	4.18	4.57	7.24	3.22	3.77	5.10	5.18
	Charpy, ft-lb. per inch of notch	5.68	5.18	5.29	6.57	4.23	4.72	5.14	4.94

[a] Tentative Specifications for Cellulose Acetate Molding Compounds (D 706 – 44 T), 1944 Book of A.S.T.M. Standards, Part III, p. 1549.

for the medium acetyl products are plotted in Fig. 17.

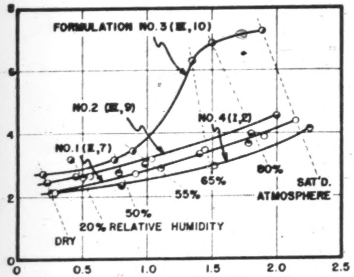

FIG. 17.—Effect of Relative Humidity on Impact Strength of Injection-Molded Cellulose Acetate. (Medium-Acetyl Formulations.)

To obtain information on the effect of weathering, tension specimens of formulations Nos. 2 and 6 were exposed at 45 deg. facing south in the open at Springfield, Mass., and Miami, Fla. Tensile properties were determined at 2-month intervals, and the results summarized in Table X.

TABLE X.—EFFECT OF WEATHERING ON TENSILE PROPERTIES; INJECTION-MOLDED CELLULOSE ACETATE.

Duration of Exposure to Weather	Location of Exposure	Tensile Yield Stress, psi.		Elongation at Break, per cent		Water Content When Tested, per cent	
		Formulation No. 2, A.S.T.M. Type III, 9	Formulation No. 6, A.S.T.M. Type IV, 15	Formulation No. 2, A.S.T.M. Type III, 9	Formulation No. 6, A.S.T.M. Type IV, 15	Formulation No. 2, A.S.T.M. Type III, 9	Formulation No. 6, A.S.T.M. Type IV, 15
0	...	3510	3330	28	44	1.05	0.82
2 months	Springfield, Mass.	3820	3730	22	23	1.36	1.00
	Miami, Fla.	4200	4070	18	24	1.42	1.00
4 months	Springfield, Mass.	4110	3970	18	29	1.34	1.00
	Miami, Fla.	3670	3780	7.7	11	1.49	1.04
6 months	Springfield, Mass.	4110	3970	16	22	1.43	0.93
	Miami, Fla.	3770	3620	2	3	1.45	1.07

DISCUSSION

The general effects of temperature and humidity on the mechanical properties of organic plastics have been summarized in a previous paper (4), and detailed data of this type for cellulose acetate and cellulose nitrate sheet plastics have been reported separately (3). The results of the present study present no contradictions to the earlier data and the same general conclusions hold throughout.

The behavior of injection-molded cellulose acetate in tension changes markedly with changes in temperature, and the character of these effects is shown clearly by the stress-strain curves (Fig. 1). In general, yield stress, breaking strength, and modulus of elasticity increase as the temperature is reduced and decrease as it is raised. Proportional limit changes in the same fashion (Fig. 2). The degree of plasticization influences the strength at any temperature but not the shape of the temperature-strength or temperature-modulus curve (Figs. 4, 5, and 8).

Elongation at break is greatly influenced by temperature also, and generally decreases as the temperature is lowered and increases as temperature is raised. However, a retrogression occurs above about 50 C. (122 F.), above which temperature these materials begin to become "weak" (Figs. 6 and 7). Ductility (elongation between yield point and break) follows the above trends also and disappears entirely somewhere between 0 C. (32 F.) and −25 C. (−13 F.). Below this point rupture takes place before a yield point is reached. This behavior is a good illustration of the effect cited by Carswell and Nason (**4**, p. 23), the point of rupture moving back toward the origin along the generalized stress-strain curve as the temperature is reduced. As ductility decreases, toughness as measured by impact tests would be expected to decrease also, and this is shown to be the case (Fig. 16).

Plasticizer formulation appears to have more influence on elongation than it does on strength, with respect to temperature effects. The inclusion of aromatic phosphate ester appears to improve ductility at elevated temperatures (Figs. 6 and 7). As might be expected, an increase in plasticizer content, of any given type, improves elongation at all temperatures, although the differences are small at low temperatures.

The effect of temperature on flexural strength follows the same trends as on tensile yield stress.

The impact strength of injection-molded cellulose acetate is very greatly influenced by changes in temperature, as would be expected from examination of the stress-strain curves. Impact resistance falls off rapidly below about 20 C. (68 F.) and levels off to values in the range of 0.28 to 0.50 ft-lb. per inch of notch at temperatures below about −10 C. (15 F.). Impact strength at any temperature tends to increase with increase in plasticizer content, although the differences are negligible at temperatures below about −20 C. (−4 F.).

Moisture acts as an efficient plasticizer for cellulose derivatives, and the properties of cellulose acetate plastics are influenced consideraby by the amount of moisture present. This amount depends upon the relative humidity of the ambient atmosphere at any given temperature.

Examination of the stress-strain curves of Fig. 3 shows that yield stress, breaking strength, and modulus of elasticity are increased by reduction in humidity and *vice versa,* whereas elongation is decreased by reduction in humidity and increased by increase in humidity. Figures 9 and 10 show how yield stress varies with humidity and the relation between humidity and moisture content of the specimen. In general, as plasticizer content is increased, moisture absorption at any relative humidity level is decreased. However, the general nature of the yield stress-moisture content curves seems to be independent of plasticizer content. The same trends are evident with respect to modulus of elasticity (Fig. 13) and flexural strength (Fig. 15). It also is interesting to note that the formulations from high-acetyl content cellulose acetate absorb less moisture at any given humidity than do the medium-acetyl formulations, but effects of variations in humidity on strength properties follow exactly the same trends, qualitatively and quantitatively (Figs. 9 and 10). Phosphate ester in the plasticizer formulation tends to decrease moisture absorption slightly, especially at very high humidities (Figs. 9, 10 11, 15, and 17).

The plasticizing action of moisture is quite evident in the impact data (Fig. 17); toughness, as measured by this test, increases appreciably as humidity is increased.

The above data should be utilized when designing parts of cellulose acetate for applications which involve exposure to temperatures or humidities appreciably different from normal.

The effect of exposure to the weather on the strength properties of cellulose acetate and cellulose nitrate sheet plastics has been described in a previous publication (**5**). The data herein presented (Table X) show that injection-molded cellulose acetate formulations resemble cellulose acetate sheet plastics in that prolonged weathering impairs ductility and toughness. It is possible to retard this deterioration markedly by the incorporation of special plasticizers in the composition (6), and such formulations should be chosen for applications where direct outdoor exposure for long periods of time is involved.

Acknowledgment:

We are indebted to Charles Trudeau and David Telfair of the Research Department, Monsanto Chemical Co., Plastics Div., Springfield, Mass., for assistance in the experimental work.

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EFFECT OF WEATHERING ON INSULATION RESISTANCE OF PLASTICS*

By Leslie H. Campbell,[1] Arthur H. Falk,[1] and Robert Burns[1]

Synopsis

The deleterious effect of sunlight on the insulation resistance of hard rubber and other of the older insulators is well known. The purpose of this paper is to furnish similar information on several of the plastics of commerce which are finding increasing usage in the electrical industries.

When a new material is proposed for use in communication apparatus, one of the first tests is for insulation resistance. If it is not a good insulator its availability for precision electrical equipment is seriously restricted. Direct-current insulation resistance may not even be of prime importance in many applications; for example, high dielectric strength may be paramount. Nevertheless, its chances of being adopted for general use in the communication field are rather slim if it is poor in insulation resistance. In addition, the insulation resistance test is an extremely delicate measure of the presence of moisture, its distribution and probable effect on many other properties.

The specimens were given the standard humidifying treatment, measured for insulation resistance, then exposed to the weather. After suitable exposure, the samples were re-humidified and re-measured. The separate effects of direct exposure, as well as exposure under glass, are given. Data on the degrading power of light alone, and heat alone, are presented.

Since most communication apparatus is designed for long life, and cannot be kept in darkness or entirely out of the weather, the data suggest discretion in the choice of plastics. As a class they are unusually good, in fact, unexcelled. If properly chosen and engineered, they will serve the electrical engineer well.

With the adoption of phenolics for electrical applications, it became desirable to determine the effect of aging and weathering on such electrical properties as insulation resistance. Consequently, since 1927, samples of phenolic moldings were exposed on the roof of the Laboratories' building, New York City, to (1) outdoors, with no protection from rain, dust, or other atmospheric influences, and (2) outdoors, but in a cabinet protected with glass doors. These early tests, confined to general purpose phenolics, indicated deterioration in insulation resistance not entirely explainable by cumulative absorption of moisture.

Early in 1939, test bars of polystyrene, methyl methacrylate, vinyl chloroacetate, and cellulose acetate butyrate were exposed in the glass-covered cabinet. Early in the summer of 1945, after 6-yr. exposure, the insulation resistance of these samples was determined, first as removed from the roof cabinet, and next after exposure to 90 per cent relative humidity at 85 F. for 4 days. The averaged insulation-resistance values expressed in megohms (1,000,000 ohms) are given in Table I.

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

[1] Bell Telephone Laboratories, Inc., New York, N. Y.

Subsequent to 1939, test specimens of mica- and cellulose-filled phenolics and asbestos-filled melamine were exposed outdoors in both a southern urban (Miami, Fla.) environment and in a northern industrial (New York, N. Y.) environment for periods of six and eighteen months, respectively. These exposures showed that under high humidities, substantially lowered insulation resistance resulted, with the mica-filled phenolic and asbestos-filled melamine suffering severe degradation. (See Table II.)

TABLE I.—EFFECT OF WEATHERING ON INSULATION RESISTANCE.
Values in megohms.

Material	As Taken from Roof Cabinet	After Humidification
Polystyrene	>203 000	59
Methyl methacrylate	>203 000	38 500
Vinyl chloroacetate	>203 000	6
Cellulose acetate butyrate	>203 000	8 350

TABLE II.—EFFECT OF WEATHERING ON INSULATION RESISTANCE.
(All results after 4 days at 90 per cent relative humidity, 85 F.)
Values in megohms.

Material	Not Weathered	Florida, 6 months	New York, 18 months
Phenol-cellulose	2 650	222	100
Phenol-mica	5 650	4	7
Melamine-asbestos	>200 000	1	4

TABLE III.—EFFECT OF RELATIVE HUMIDITY ON WEATHERED SPECIMENS.
Values in megohms.

Material	15% R. H.	50% R. H.	65% R. H.	75% R. H.	90% R. H.
Phenol-cellulose:					
Florida exposed	48 000	21 000	2 810	657	222
New York exposed	9 000	8 000	4 520	470	100
Phenol-mica:					
Florida exposed	>200 000	63 500	214	49	4
New York exposed	>200 000	81 500	748	38	7

NOTE: Samples were dried at 120 F. for 24 hr. between humidity exposures.

To determine the relationship of degradation to various humidities, the phenolic specimens were tested after conditioning 4 days as shown in Table III.

These data indicated the necessity for a more comprehensive study of the effect of weathering on the insulation resistance of commercial molding compounds which might be considered for electrical applications. Such an investigation obviously must cover both the thermosetting and thermoplastic molding compounds.

SPECIMENS AND PROCEDURES

Specimens for the most part were 4 by 7 by $\frac{1}{8}$-in. panels, either molded or cut from commercial sheet. They were prepared in accordance with A.S.T.M. Standard Methods of Test for Insulation Resistance of Electrical Insulation Materials (D 257 – 45),[2] Fig. 1. Two rows of five holes each provided nine reading positions for each specimen.

The test procedure consisted of exposing the specimen to 90 per cent relative humidity at 85 F. for 96 hr., measuring the insulation resistance, placing in the exposure location for the period chosen, rehumidifying and re-measuring. Insulation resistance was measured in accordance with A.S.T.M. Methods D 257 – 45 given above and reported in megohms. Two natural environments were selected: (*a*) outdoors at 45 deg. facing south, and (*b*) outdoors in a glass-covered cabinet fitted with ventilating louvres and positioned 45 deg. facing south. The glass was ordinary window glass and was periodically cleaned. The location was the roof of the Laboratories' building in New York, N.Y. The exposures were usually started in mid-winter and the exposure intervals varied from 2 weeks to 2 months.

To examine causative agents of degradation, selected materials having good, fair, and poor weather resistance in the natural environments were exposed in two artificial environments: (*a*) oven at 160 F., and (*b*) closed twin-arc weathering machine using light only which gave panel temperatures of approximately 130 F. Specimens and procedures were the same as for the natural environments except that the exposure intervals varied from 4 to 14 days.

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[2] 1945 Supplement to Book of A.S.T.M. Standards, Part III, p. 57.

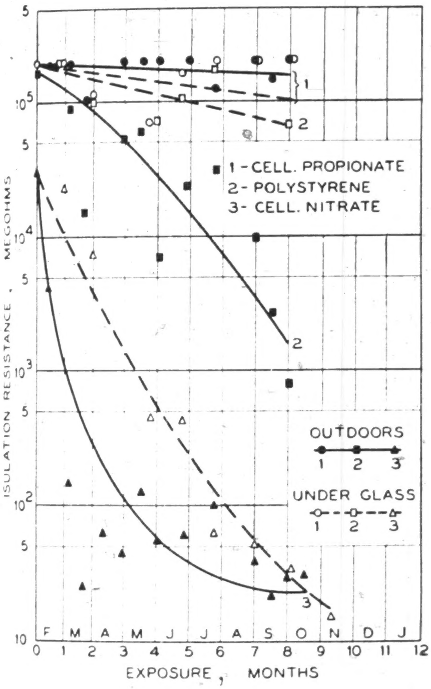

FIG. 1.—Change in Insulation Resistance of Thermoplastic Resins with Exposure.

MATERIALS AND TEST DATA

Table IV lists the materials tested in the natural environments and gives the data obtained after 2 and 6-months' exposure as taken from the generalized curves.

Table V lists the materials tested in the artificial environments and gives the data obtained after 2 weeks' and 2 months' exposure as taken from the generalized curves.

DISCUSSION

Figure 1 shows deterioration in insulation resistance with exposure both outdoors and under glass three thermoplastic materials—cellulose propionate, polystyrene, and cellulose nitrate. These materials were rated respectively as excellent, good, and poor and are representative of the course of deterioration. The first two were compression-molded and the third was prepared from commercial sheet. The exposure periods vary from 6 to 9 months.

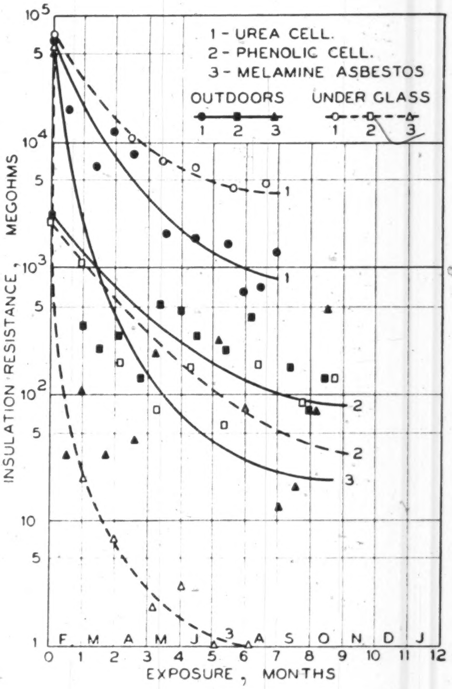

FIG. 2.—Change in Insulation Resistance of Thermosetting Resins with Exposure.

Figure 2 shows relative rates of deterioration for the thermosetting resins, urea-cellulose, phenol-cellulose, and melamine-asbestos, materials rated respectively fair, poor, and very poor. All materials were compression-molded.

Figure 3 shows the deterioration in insulation resistance of polystyrene with

TABLE IV.—CHANGE OF INSULATION RESISTANCE WITH EXPOSURE TO NATURAL ENVIRONMENTS.

Materials	Insulation Resistance, megohms					
	Outdoors			Under Glass		
	Initial	After Exposure		Initial	After Exposure	
		2 Months	6 Months		2 Months	6 Months
THERMOPLASTIC RESINS						
CELLULOSE PROPIONATE						
Pigmented-Ivory	>194 000	190 000	170 000	>194 000	170 000	125 000
CELLULOSE ACETOBUTYRATE						
Type H6—Clear	115 000	17 000	1 000	160 000	160 000	125 000
Type H5—Black	165 000	110 000	36 000	180 000	160 000	130 000
Type S4—Clear	>194 000	170 000	130 000	>194 000	170 000	130 000
Type S2—Black	>194 000	170 000	125 000	>194 000	170 000	125 000
ETHYL CELLULOSE						
Pigmented Black	>194 000	160 000	40 000	>194 000	170 000	90 000
METHYL METHACRYLATE						
General Purpose—Clear	>194 000	15 000	430	>194 000	180 000	140 000
General Purpose—Clear	>194 000	120 000	9 500	>194 000	180 000	75 000
General Purpose—Clear Cast	>194 000	180 000	60 000	180 000	190 000	>194 000
General Purpose—Red Translucent	>194 000	125 000	38 000	>194 000	100 000	15 000
Heat Resistant—Clear	>194 000	155 000	20 000	>194 000	170 000	55 000
POLYSTYRENE						
General Purpose—Clear	180 000	85 000	6 900	>194 000	175 000	98 000
Heat-Resistant—Copolymer	>194 000	75 000	4 500	>194 000	165 000	65 000
CELLULOSE ACETATE						
Type H4—Clear	50 000	19 000	3 000	50 000	43 000	32 000
Type H4—Black	23 000	15 000	7 000	23 000	20 000	14 000
Type S—Black	9 000	8 800	6 800	9 000	10 000	9 800
POLYDICHLOROSTYRENE						
Heat Resistant—Clear	>194 000	24 000	1 000	>194 000	170 000	85 000
POLYETHYLENE						
Amber Translucent	92 000	70 000	1 700	>194 000	175 000	140 000
POLYVINYL CHLOROACETATE						
Clear	125 000	27 000	2 800	130 000	120 000	53 000
Black	140 000	125 000	36 000	115 000	24 000	2 300
ESTER-STYRENE COPOLYMER						
Cast (Thermosetting)	28 000	5 600	900	28 000	20 000	12 000
VINYLIDINE CHLORIDE						
Amber Sheet	2 500	5 800	430	330	1 700	260
CELLULOSE NITRATE						
Sheet—Clear	10 900	280	28	10 900	3 700	110
Sheet—Clear	1 500	140	32	1 500	1 700	230
POLYVINYL CHLORIDE						
Electrical Grade	56	160 000	....	280	2 300	....
HARD RUBBER						
Natural	>194 000	8 800	....	190 000	3 200	...
Buna S	21 000	47	....	19 000	28	...

TABLE IV.—*Concluded*

Materials	Insulation Resistance, megohms					
	Outdoors			Under Glass		
	Initial	After Exposure		Initial	After Exposure	
		2 Months	6 Months		2 Months	6 Months
THERMOSETTING RESINS						
UREA FORMALDEHYDE						
Cellulose-Filled—White	62 000	7 200	960	70 000	14 000	4 000
ANALINE FORMALDEHYDE						
Mica-Filled—Brown	180 000	1 300	24	190 000	75	30
Mica-Filled—Brown	520	95	36	560	120	38
Mica-Filled—Brown	5 000	290	68	7 000	380	90
PHENOL FORMALDEHYDE						
Unfilled—Red	9 800	600	28	8 800	1 300	120
Cellulose-Filled—Black	2 700	720	125	2 600	580	70
Cellulose-Filled—Black High Resin Content	13 000	3 800	540	3 000	1 100	270
Rag-Filled—Black	41	22	12	64	25	8
Asbestos-Filled—Black	1 200	150	35	10 000	260	6
MELAMINE FORMALDEHYDE						
Cellulose-Filled—White	1 400	1 000	650	9 800	6 500	3 100
Asbestos Filled—Grey	>194 000	920	3	>194 000	23	2
Asbestos-Filled—Grey	58 000	400	30	68 000	7	1

TABLE V.—CHANGE OF INSULATION RESISTANCE WITH EXPOSURE TO ARTIFICIAL ENVIRONMENTS.

Materials	Insulation Resistance, megohms					
	Oven at 160 F.			Light at 130 F.		
	Initial	After Exposure		Initial	After Exposure	
		2 Weeks	2 Months		2 Weeks	2 Months
THERMOPLASTIC RESINS						
METHYL METHACRYLATE						
Heat Resistant—Clear	>194 000	194 000	>193 000	>194 000	16 000	1 500
General Purpose—Pigmented White	>194 000	194 000	>193 000	>194 000	18 000	970
POLYSTYRENE						
General Purpose—Clear	>194 000	194 000	>193 000	>194 000	3 500	260
THERMOSETTING RESINS						
UREA FORMALDEHYDE						
Cellulose-Filled—White	>194 000	198 000	140 000	>194 000	55 000	16 500
ANALINE FORMALDEHYDE						
Mica-Filled—Brown	>194 000	170 000	120 000	>194 000	1 000	54
Mica-Filled—Brown	>194 000	3 800	150	>194 000	140	10
Mica-Filled—Brown	>194 000	80 000	9 200	>194 000	5 000	130
Unfilled—Red	>194 000	193 000	193 000	>194 000	230	23
PHENOL FORMALDEHYDE						
Cellulose-Filled—Black	1 100	3 800	8 000	850	1 200	470
Unfilled—Red	10 800	21 000	54 000	6 500	8 100	2 200
MELAMINE FORMALDEHYDE						
Asbestos-Filled—Grey	11 300	17 000	950	24 800	43	2
Unfilled—Clear	8 800	50 000	180 000	37 000	1 400	180

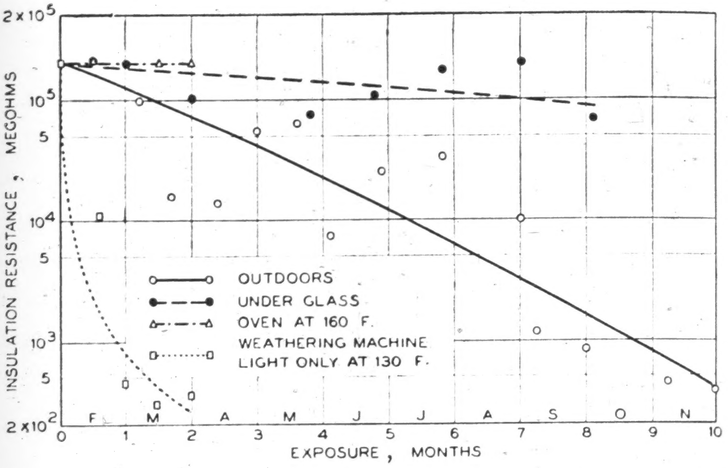

FIG. 3.—Change in Insulation Resistance with Natural and Artificial Exposure of Polystyrene.

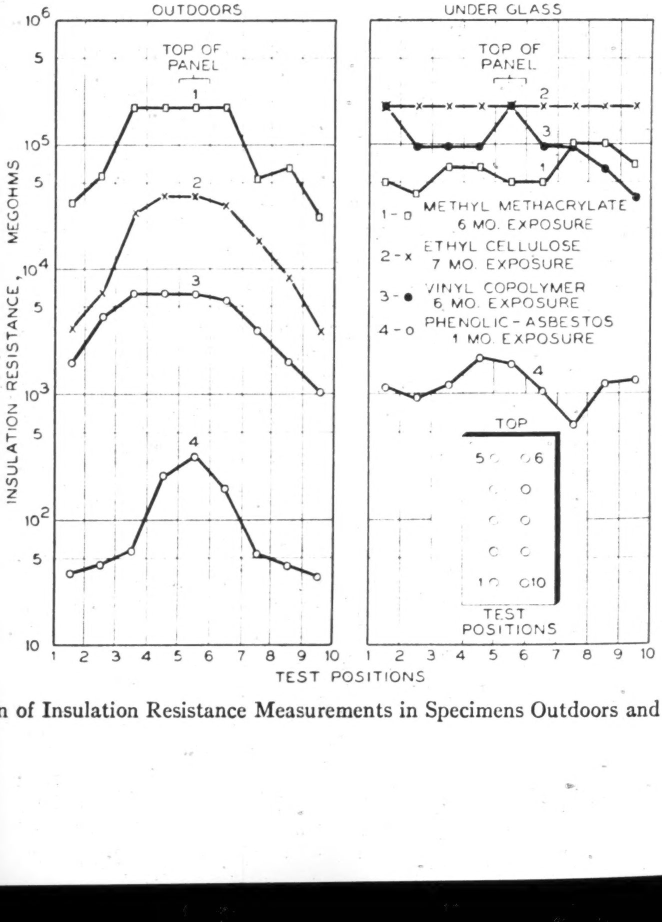

FIG. 4.—Pattern of Insulation Resistance Measurements in Specimens Outdoors and Under Glass.

exposure in the outdoor, under-glass, oven, and light environments. It is fairly typical of the unfilled resins examined. In many of the filled thermosetting materials the insulation resistance increased after oven aging. Analine and melamine-filled resins, however, did decrease, but not in a manner comparable to the light-exposed specimens.

Figure 4 shows the typical pattern of insulation-resistance readings over the area of the panel after outdoor exposure, the test positions on the top part of the specimen showing considerably higher insulation resistance. The under-glass specimens showed little of this, presumably because the washing action of rain and melting snow was absent.

The initial insulation resistance of plastic materials after conditioning in 90 per cent relative humidity for 4 days varied from 15 megohms for rag-filled phenolic to greater than 200,000 megohms for cellulose propionate. Most, however, were 10,000 megohms or more, many being in the 200,000 megohm range, while some few had initial values below 1000 megohms. For the most part, two specimens of the same plastic had the same or similar values, but in a few cases considerable variation existed.

The question arose as to the effect on the initial insulation of the smoothness of the surface of the specimen and also if iron molds exerted any catalytic effect during the molding process which might contribute to lower insulation, particularly in reference to the chlorinated plastics. Accordingly, phenol formaldehyde with cellulose filler and polyvinyl chloride acetate were molded with mold face plates having the following surfaces, (*a*) rough steel, (*b*) polished steel, (*c*) rough chromium plate, and (*d*) polished chromium plate. Insulation readings on these specimens taken after the usual conditioning showed little or no difference among the various surfaces. These specimens were not exposed specimens of two samples of c calendered vinyl copolymer sheet, with polished and one with a matte surface, were measured and exposed for 96 hr. in the weathering machine, light only. The two differed little in initial insulation resistance and also with exposure, the polished specimen declining from 181,000 to 110 megohms, and the matte from 200,000 to 530 megohms. In view of the sensitiveness to light of this material, the surface influence does not appear critical.

The averaged readings of the nine positions obtained after each exposure, as interval followed interval, had a rather wide oscillatory pattern in their variation from the generalized curve. In some cases the outdoor specimen showed the wider oscillation and in some cases the under-glass panel. In all cases, though, the trend was downward. Whether this oscillatory effect can be considered characteristic of plastics in general, or is due to the methods employed was not determined.

The soft grades of the cellulose esters, that is, those containing plasticizers, showed less deterioration than the hard grades. Some of the materials exhibited an increase in insulation resistance during the first few exposure intervals and in the case of some of them, notably vinyl chloride and vinylidine chloride, this persisted for months.

The relative rates of deterioration by the two environments, outdoors and under-glass, was, in general, either markedly higher for outdoors or similar for the two.

Pigmented and dyed thermoplastics had, in general, the same deterioration rate as the clear of the same material. Further exposure may reveal some differences, a possibility suggested by the behavior of paints, the clears of which fail markedly faster than pigmented

1471 Check

enamels. Not a few performance analogies to paint products have appeared in this and other exposure work on plastics.

In the weathering machine environment, deterioration took place which was similar in type and degree to that experienced in outside and under-glass exposure, though in most cases the rate was faster. The ratio of exposure for comparable degradation in the weathering machine and in the natural environments showed some wide extremes, but for most of the materials the ratio was 2 months in the weathering machine to 5 months outdoors and 6 months or more under glass. Exposures in the oven at 160 F. gave results that had only minor agreement with those obtained in the natural environments; only in the analine and melamine resins containing fillers did any deterioration take place in 2 months' exposure.

That the lowering of insulation resistance with weathering is, with some materials, exclusively a surface phenomenon is illustrated by the behavior of mica-filled phenolics and asbestos-filled melamines when removed from a moist atmosphere to a dry atmosphere. A value of a few megohms under 90 per cent relative humidity will increase to many thousands of megohms in a few seconds.

To examine the products of degradation, exposed specimens of phenol-rag, phenol-asbestos, melamine-asbestos, and cellulose nitrate were used. These specimens had been exposed approximately 9 months in the natural environments and the insulation resistance had dropped to 50 megohms or less. They were treated in two consecutive steps as follows: (1) Completely immersed in 2 liters of water for 24 hr., sopped dry (no rubbing), dried at 120 F. for 48 hr., reconditioned in humidity in the usual way, and measured; and (2) disassembled, polished with mild liquid-abrasive polish (designed to remove chalked material on the surface but not cut into the plastic), washed with 5 per cent ammonia water, reassembled, dried for several days at room conditions, conditioned in humidity, and measured. The soaking treatment restores the insulation considerably in all specimens and markedly more in the under-glass specimens than in the outdoor ones. The polishing either made no further change or restored the value still more in all materials except the outdoor specimen of melamine-asbestos. In polishing as in soaking, the under-glass specimens showed more marked recovery, cellulose nitrate greatly exceeding its initial value.

CONCLUSIONS

1. Molded plastic materials differ widely in their initial insulation resistance and in the rate and the degree to which this insulation deteriorates with exposure.

2. The relative rate of deterioration in the two environments, outdoors and under glass is, in general, either markedly higher for outdoors or similar for the two.

3. Pigments and dyes in thermoplastics produce little or no change in either initial insulation resistance or in the rate of deterioration, as compared to the clear resin, though plasticizers in the cellulose esters appear to have an inhibitive effect. For the thermosetting materials, fillers, dyes, etc., may increase the initial value somewhat but have small or no effect on the rate and degree of deterioration, as compared to the unfilled resin.

4. With respect to artificial accelerated environments, preliminary data indicate that such methods involving light may have promise for predicting the gross behavior of plastics in natural

exposure. Of the two factors, light with its accompanying heat and heat alone, light appears to be by far the more effective.

5. The products of degradation which cause lowered insulation resistance of phenol and melamine formaldehyde and cellulose nitrate are largely water soluble.

Acknowledgments:

The authors wish to express their thanks to Anne S. Wrubel and George Deeg for assisting in this work.

DISCUSSION

Mr. J. A. Loranger.[1]—Mr. Campbell, from your paper I gather that you consider current leakage, as measured by the insulation resistance test, a surface phenomenon.

In laminated plastics, it has been my experience that the filler used has considerable bearing on the insulation resistance of the laminate. Based on this experience, I have always felt that the property in question was used as a measure of surface plus a substantial internal current leakage for a specified voltage. Now am I correct in this deduction; and if so, what is the ratio of the surface to internal current leakage as measured by the Standard Methods of Test for Insulation Resistance of Electrical Insulating Materials (D 257 – 45).[2]

Mr. Leslie H. Campbell (*author*).—We feel, rather, that most of the leakage is at or near the surface. What is actually measured, however, is surface plus volume leakage. But the leakage through the volume of most plastic materials, I believe, has been proven by others to be very small when using this short-time test. Most plastics in the cross sections used here take more than four days to absorb sufficient moisture to affect substantially the volume resistance. But the data actually include volume as well as surface leakage.

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[1] Plastics Engineer, Radio Div., Sylvania Electric Products, Inc., Warren, Pa.

[2] 1945 Supplement to Book of A.S.T.M. Standards, Part III, p. 57.

WATER DETERMINATION IN NEW AND USED INSULATING OILS BY DOBLE METHOD*

By Frank C. Doble[1]

Synopsis

The significance of a measurement of the water content of an insulating oil is briefly outlined in this paper as a preface to a detailed description of the Doble apparatus and method.

Designed and developed to meet a long-felt need in the art of preventive maintenance, its use extends the scope of investigations of used oils which are badly contaminated and dark colored and facilitates the general study of the insulating oil problem. For clarity of description, the author makes use of a nomenclature in terms of known electrical values expressed as high-loss water and low-loss water to obviate the difficulties attendant to the use of chemical or physical terms, the application of which may be controversial.

The apparatus and method, which are fully disclosed in the paper, extract water from a sample of oil by heating, shaking and vacuum, making use of a newly developed filter to remove unwanted contaminants and permit the water alone to be absorbed in a tube of P_2O_5 for weight determination. It combines advantages of speed, accuracy and ease of operation.

Since water has long been recognized as one of the worst enemies of electrical insulation, the determination of the water content of insulating oil is essential to efficient preventive maintenance of electric power equipment and valuable in the development of new oils. The apparatus and method described in this article are the result of several years' search for a means to measure accurately the water content of used oils that are dark colored or badly contaminated, as well as to measure the water content of partly deteriorated or new oils.

It appears that water may be present in insulating oils in different physical or chemical states or conditions; for example, in solution, suspension, or some finely divided intermediate form. A search of the literature[2] indicates that the physicists and chemists are not in agreement as to the precise nature of the various states or conditions in which water exists, and it may not be possible to distinguish them separately and hence measure them accurately in practice.

Even though some of these conditions of water cannot readily be differentiated by known simple chemical or physical measurements, the effect of their presence in insulating oils can be expressed in terms of electrical measurements.

Long experience with every-day operation of electric power insulation demonstrates conclusively that there are two distinctly different electrical effects which

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24-28, 1946.

[1] President, Doble Engineering Co., Medford, Mass.

[2] N. E. Dorsey, "Properties of Ordinary Water Substances," First Edition, Reinhold Publishing Co., New York, N. Y., pp. 161-177 (1940).

can be brought about by the presence of water in oil. For example, it is well known that, under certain conditions, as little as 10 to 20 ppm. of water in oil will greatly reduce its dielectric-breakdown value and constitute an immediate hazard which may even cause a failure in the operation of the high-voltage equipment with which it is associated. It is also well known that, under other conditions, several hundred parts per million of water may be present in an insulating oil without significantly reducing its electrical insulating value but, nevertheless, it may be a potential hazard.

When the well-known dielectric breakdown test is used to study moisture conditions in oil, it probably detects only the presence of water-in-oil conditions that cause the immediate hazard referred to above. It gives little or no indication of any other water-in-oil conditions that may be present.

In order to describe and study moisture conditions in oil, it has been the custom of the Doble Co. to use the term "low-loss" for water which is electrically inactive and "high-loss" for water that is electrically active regardless of physical form or chemical properties. Although the line of demarcation between high-loss and low-loss water varies with conditions, such as temperature, it is possible to set up a readily reproducible value as a standard for a given condition.

For this purpose, a dielectric-loss or power-factor measurement can be used as an index to differentiate between high-loss and low-loss water with a base line selected at will to meet a desired condition.

Water in oil is considered to be in a low-loss state when a measurement of its dielectric loss and power factor shows readings below reference values which arbitrarily have been selected by experience to represent a condition that is harmless or safe electrically, that is, not detrimental to the high-voltage insulating property of the oil, within practical limits. Conversely, when the measured values of the dielectric loss and power factor are greater than the reference values already mentioned, the term high-loss applies.

It should be borne in mind that the use of the terms high-loss and low-loss water is not intended to describe any physical or chemical form of the water itself, but rather an electrical state or condition which exists due to the presence of water in oil.

Thus defined, the terms high-loss and low-loss will be used to explain the need for and significance of water determination in new and used oils. Since the use of an electrical test indicates only the presence of the high-loss water that is an immediate hazard, the complete study of the effects of water conditions in oil requires some knowledge of the total amount of water present.

The determination of the water content of insulating oil is essential because:

1. The presence of high-loss water constitutes an immediate electrical hazard. The amount of high-loss water can be determined only by some extraction means.

2. The presence of low-loss water in oil may be a potential hazard even though temporarily it does not reduce the dielectric value of the oil. A small drop in temperature may cause some of the low-loss water to become high-loss water, which is an immediate failure hazard.

3. Much of the moisture that enters and becomes a hazard to solid insulation immersed in oil is transferred from the oil in a low-loss form.[3]

4. Preliminary studies indicate that the total amount of low-loss water that

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[3] F. M. Clark, "Water Solution in High-Voltage Dielectric Liquids," *Transactions*, Am. Inst. Electrical Engrs. Vol. 59, August, 1940, p. 433.

an oil can tolerate when saturated may be highly significant in the study of oil deterioration.

5. Control of the water-retention characteristics in the development of new oils is important.

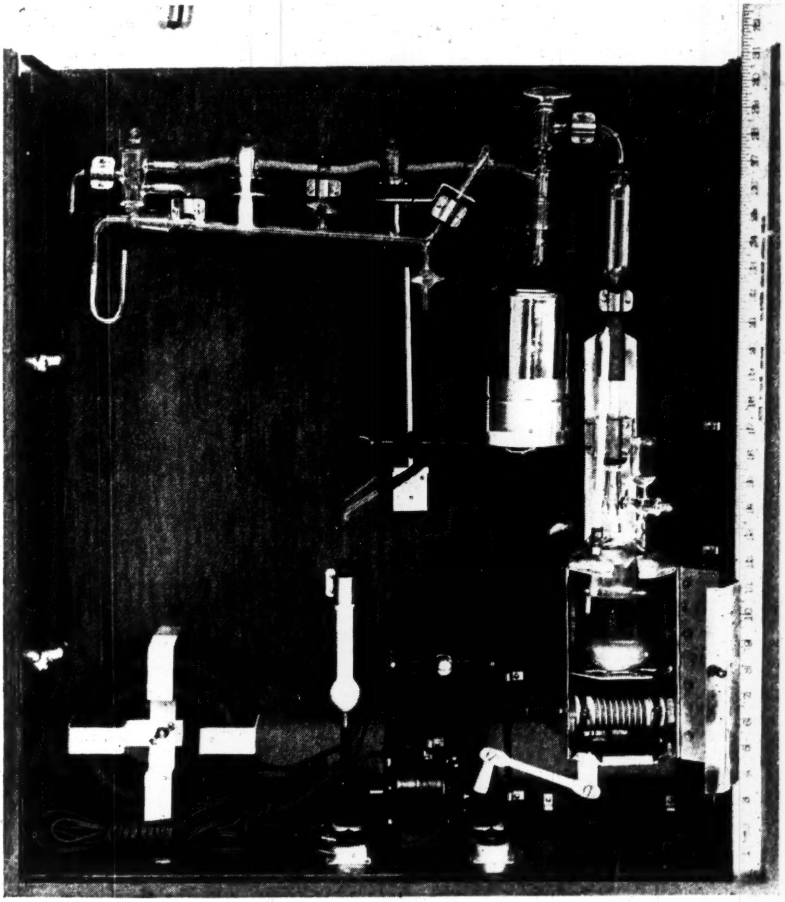

FIG. 1.—Photograph of Doble Water-Extraction Apparatus.

The apparatus and method described in this paper are designed to measure accurately the total water content of both new and used oils. Particular emphasis has been put on the solution of the more difficult part of the water measurement problem, namely, the testing of old oils which are dark in color, contain dissolved sludge, and may have dissolved in them various substances derived from deteriorated solid insulation, or from varnishes and chemical substances used in the manufacture of the apparatus which is immersed in the oil. As some of these substances are chemically active, the employment of a chemical test for water is always subject to the possibility of side reactions which may give a false indication of water content. To test an oil for the presence of substances which might react with the reagent to be used in the water test would be time-consuming and expensive. The ideal water test, accordingly, is a quick test which side-steps this difficulty, is easy to make, and requires a minimum of special training on the part of the operator. The ability to reproduce results is of course a prime requisite of any quantitative test. In the literature there is a reference[4] describing the Rodman method which utilizes the basic functions of heating, shaking, and evacuation in conjunction with an absorption tube containing phosphoric anhydride. This method was subject to errors, when applied to deteriorated oils, arising from reactions between the phosphoric anhydride and the substances in the distillate from the oil sample other than water.

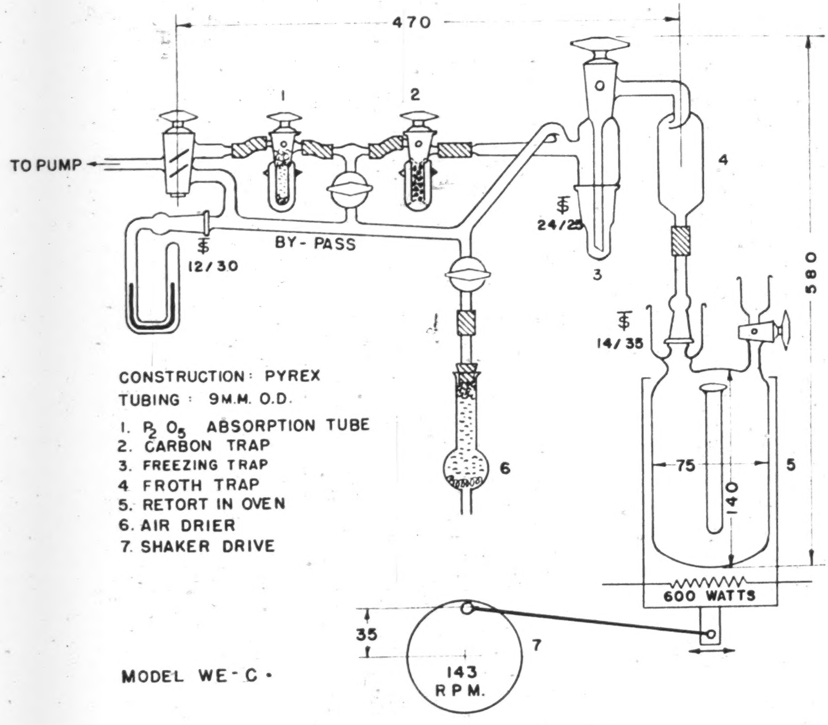
470
TO PUMP
1
2
BY-PASS
12/30
24/25
3
4
580
14/35
CONSTRUCTION: PYREX
TUBING: 9M.M. O.D.
1. P_2O_5 ABSORPTION TUBE
2. CARBON TRAP
3. FREEZING TRAP
4 FROTH TRAP
5. RETORT IN OVEN
6. AIR DRIER
7. SHAKER DRIVE
6
75
140
5
600 WATTS
35
7
143 RPM.
MODEL WE-C

FIG. 2.—Schematic Diagram of Apparatus in Fig. 1.
Dimensions are in millimeters.

In the Doble method, water is extracted from the oil by heating, shaking, and vacuum; light ends and contaminants are removed by means of a filter of activated carbon; and the true amount of water is determined from its weight as absorbed in a tube of P_2O_5. The water content is then calculated from the weights of the sample and the extracted moisture.

Fundamental Processes and Observations:

The Doble system is shown in Figs. 1 and 2. The train is constructed of pyrex glass with the exception of rubber or neoprene seals which guard the joint between the oscillating retort and the fixed portion of the train which serve as connections for the absorption tubes.

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[4] C. J. Rodman, "Determination of Moisture in Insulating Oils," *Industrial and Engineering Chemistry*, Vol. 13, pp. 1149–1150 (1921).

In operation, the system is evacuated, and a weighed sample of oil is injected into the retort. Since a partial degasification occurs during the injection and a significant portion of the moisture escapes from the oil with the dissolved air, the retort is isolated during the injection. Next, the gas is permitted to flow slowly through the freezing trap where the water congeals. Then with the gas flow directed through the filter and the absorption tube, the retort is heated and the shaking motion started. When extraction has been accomplished, the retort is isolated by a stopcock and the first step in the process is complete.

The freezing mixture is removed from around the trap and gradually the trap is heated to dryness. Much of the moisture in the trap sublimes during the early stages of the heating, but as light ends of oil and whatever volatile impurities existed in the sample are also present in the trap, sufficient heat must be applied to vaporize them. A droplet of water covered with a film of oil frequently will not vaporize until a temperature of 150 C. or higher has been reached. As a bursting droplet of water sometimes sprays tiny droplets of water onto the oily surface of cooler portions of the trap, it is desirable to retrieve such particles by replacing the freezing mixture. Any free liquid will return in a few seconds to the chilled area, and a second heating is usually sufficient to secure complete vaporization.

On leaving the freezing trap, the vapors and gases flow through the carbon filter, which absorbs all hydrocarbons, and thence through an absorption trap where the water vapor reacts with phosphoric anhydride, and the air or other gases escape to the vacuum system. As ortho-phosphoric acid has an extremely low vapor pressure, the escape of extracted water to the vacuum system is negligible.

The fundamental processes involved in this extraction method are all relatively simple and well established. However, to secure the accuracy needed in a study of the function of water in insulating oil, and to provide an apparatus which is dependable and also easy to operate, an extended study of many of the reactions involved was needed.

As used oils frequently contain reactive substances in solution, the filter is necessary to protect the absorption tube from contamination and side reactions which would give a false indication of water pickup. The carbon filter effectively traps the impurities, but it also absorbs a small amount of the water vapor which passes through it. An essential feature of this system, accordingly, was the development of a technique for preparing and calibrating these filters in quantity.

The system operates under vacuum at all times. The vacuum requirement, however, is not critical, any pressure less than 0.5 mm. being satisfactory, and this may be obtained conveneintly from almost any rotary laboratory pump.

As it is impossible to render a glass system actually dry without resorting to high temperature, the Doble system is maintained in a state of semi-equilibrium by providing the train with an air drier through which air is admitted to the system when any element is to be removed for weighing or emptying. The retort is exposed to room air when it is emptied, but as it is always warm when emptied and is immediately evacuated when replaced, no error within the range of accuracy employed can be detected. To ensure stable conditions, the charge of exhausted sample is always left in the retort. A "blank" run is made on this spent oil before the first test run is made any subsequent day. Accordingly, every test run directly follows a previous run, and the condition of the walls of the apparatus remains relatively constant. As water content only is under investigation, it is unnecessary to clean the retort, any film of oil or sludge adhering to it having been previously dried.

Operation of the Doble System:

With the elements of the train in position, the whole system is pumped out through the by-pass, the retort is isolated by manipulating the appropriate stopcock, a freezing mixture is raised around the capsule of the freezing trap, and a weighed sample (200 g.) is injected into the retort. The evolved gas is now allowed to flow into the freezing trap slowly until the vacuum in the retort is restored. The three-way stopcock is reversed, and the absorption tube and filter are opened to the line. The heater is now turned on and the shaker started. The time is recorded and heating is continued until the well temperature reaches 75 C., but the shaking is continued until 10 min. have elapsed. The retort is now isolated, the freezing mixture lowered, and a small flame is applied to the capsule of the freezing trap. When the trap is dry, the freezing mixture is returned for ½ min., and then the trap is again heated to dryness. This operation requires about 5 min. The apparatus is allowed to rest in this condition with the pump operating for an additional 5 min. so that the over-all time from the start of the heating to the closing and removal of the absorption tube is just 20 min. As the calibration of the filters is based on this schedule, the over-all time must be observed.

Preparation and Calibration of Filters:

The preparation and calibration of the carbon filter tubes consist of two operations. A batch of tubes is loaded with some definite volume of activated carbon which has been previously cleaned by heating for 2 hr. *in vacuo* at 150 C., and the tubes are then subjected to a 4-hr. evacuation. Several tubes from the batch are then taken at random and are calibrated.

The second operation, the calibration, consists of subjecting the tubes individually to weighed amounts of water, and measuring the amount recovered after passage through the filters. It is our practice to calibrate the first tube with a small amount of water, say 5 mg., which would correspond to 25 ppm. if derived from a 200-g. sample of oil. The second tube would be tested with about 30 mg. which would correspond to 150 ppm. When plotted, input (abscissa) against output (ordinate), if the two points are equidistant from the line $x = y$, the calibration is considered satisfactory. If the two points vary in distance, another tube is tested with about 20 mg. From the position of the three plots it is usually possible to detect and check the erratic reading. In use on an unknown sample, the gain in weight of the absorption tube is found on the ordinate, and the corrected value is read on the abscissa.

The calibration procedure makes use of the extraction apparatus and follows the time schedule and technique regularly employed. The method consists of isolating the retort and placing a weighed amount of water, from a dropper, in the freezing trap capsule. The freezing mixture is then applied, the system is evacuated, the P_2O_5 and carbon tubes are opened to the line, a 10-min. interval (corresponding to the time allowed for the heating and shaking of a sample) is observed, and from then on the regular manipulations are carried out.

Application:

The Doble apparatus was designed specifically for use with transformer or other high-boiling-point hydrocarbon liquids. In its present form, it is not recommended for use with askarels. The accurate range is from 25 to about 600 ppm., and reproducibility is in the order of ±5 ppm. Lower readings can, of course, be obtained, but the probable error in the case of a 10-ppm. reading is in the order of 50 per cent.

As a research tool it is highly useful, since it enables the operator to handle samples over a considerable range of temperature without observing special precautions.

As judgment does not enter into the operation of the system, excellent results can be obtained by anyone capable of operating an analytical balance.

DISCUSSION

Mr. J. C. Balsbaugh.[1]—It is probably true that water is the single most important factor in determining the performance and life characteristics of electrical insulation. In oil-immersed insulation, water not only affects the electrical characteristics of the liquid itself but affects the solid insulation through transfer from the liquid. Accordingly, a simple test that can accurately measure the amount of moisture present in the liquid at any time is very important. I believe that the Doble method is satisfactory for this purpose.

While much work has been done to study the problem from a theoretical point of view, the satisfactory performance of high-voltage liquid-immersed insulation is based primarily on the correlation over long periods of time of various significant tests with performance experience. Experimental work has definitely indicated that there are many factors influencing the electrical effect of a given amount of water. Accordingly the separation of the effect of the water from a practical point of view into "low-loss" and "high-loss" terms is desirable.

Practical work on liquid insulation from the point of view of dielectric loss and breakdown requires tests at relatively high gradients and at power frequencies. It is believed that much could be learned about the performance and the relative condition of this type of insulation by power-factor measurements over wide-frequency, temperature, and gradient ranges on appropriate laboratory models. Furthermore, dielectric breakdown characteristics should be studied at high-frequency or with pulsing circuits as compared with power frequencies that have been generally used. Accelerated aging or life characteristics of insulation at power frequencies requires relatively high gradients to give a reasonable length of test. The use of high-frequency or pulsing circuits should give tests of approximately equal length of time but would permit the use of potential gradients corresponding to commercial practice. Thus the severity of the life tests would be obtained by changing the frequency of the voltage rather than the magnitude of the voltage.

Mr. E. A. Snyder[2] (*by letter*).—The author has contributed a valuable addition to methods for determination of small amounts of water in mineral petroleum oils. The apparatus is well designed and engineered and should with practice prove to be fairly easily manipulated. There are, however, several points in the author's paper which, I think, could and should be clarified or more fully developed.

At the top of page 1476 the author states "... under other conditions, several hundred parts per million of water may be present in an insulating oil...." I feel that this is a rather vague statement and that the author should more specifically illustrate with factual data as to what these conditions are, because although he states this to be a well-known fact, many readers of the paper may not know under what conditions it is possible.

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[1] Associate Professor of Electric Power Production and Distribution, Massachusetts Inst. of Technology, Cambridge, Mass.

[2] Insulating Oil Sales and Chief Engineer of Process Products, Socony-Vacuum Oil Co., New York, N. Y.

On page 1476 the author states "Although the line of demarcation between high-loss and low-loss water varies with conditions, such as temperature, it is possible to set up a readily reproducible value as a standard for a given condition." He attempts in the next paragraph to explain that a dielectric-loss or power-factor measurement can be used as an index to differentiate between high-loss and low-loss water with "a base line selected at will to meet a desired condition." This seems to be a very vague explanation of the difference between his so-called "low-loss" and "high-loss" water. Water is not the only cause of low dielectric strength or poor power factor value. Other very common contaminants also influence these two tests and it would seem desirable to have a more understandable statement as to how one can define or determine the line of demarcation between what he calls high-loss and low-loss water.

Later on page 1476, the author states that "... the terms high-loss and low-loss water is not intended to describe any physical or chemical form of the water itself, but rather an electrical state or condition which exists due to the presence of water in oil." To me, this paragraph is meaningless. What is this electrical state or condition? What is the author trying to say? Can he be more specific and give the reader a clearer picture of what he considers the difference between high-loss and low-loss water. Without a clearly expressed definition it is of course very difficult to understand the statement that he makes in a subsequent paragraph, "that high-loss water is an immediate electrical hazard."

The author states that the amount of high-loss water can be determined only by some extraction means. I should like an explanation as to what happens to the so-called low-loss water while he is extracting the high-loss water.

Under item 4 of this same paragraph, and at the top of page 1477 the author states that the "total amount of low-loss water that an oil can tolerate when saturated may be highly significant in the study of oil deterioration," yet I do not see that he has shown in his paper any means of quantitatively determining the actual amounts of both low-loss and high-loss water—or in other words, differentiating between the quantitative percentages of both types of water.

In the second column, page 1477 the author simply states that certain dissolved sludges or other contaminants or "chemical substances" are present in oil and that they are chemically active and consequently invalidate the results obtained by any chemical test for the determination of water. A statement of this kind should be substantiated by actual data. I cannot see where the author has proved this statement which indirectly infers that chemical tests such as the Karl Fischer test will not give accurate results.

MR. T. K. SLOAT.[3]—In this paper Mr. Doble states: "it is well known that, under certain conditions, as little as 10 to 20 ppm. of water in oil will greatly reduce its dielectric-breakdown value and constitute an immediate hazard," and later: "It is well known that, under other conditions, several hundred parts per million of water may be present in an insulating oil without significantly reducing its electrical insulating value." What are these conditions? I do not feel that these conditions are well known.

Mr. Doble assumes there is a line of demarcation between the so-called "high-loss" and "low-loss" water. What is the basis for establishing a line of demarcation when in the following paragraph he states that a base line can be selected at will to meet a desired condition?

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[3] Westinghouse Electric Corp., Sharon, Pa.

On page 1476 Doble says that "The amount of high-loss water can be determined only by some extraction means." What happens to the low-loss water in such a test? In Item 2, a drop in temperature is given as the cause for some of the low-loss water becoming high-loss water. This seems to be questionable since a decrease in temperature would more than likely improve the power factor and this would be the only means of detecting any change in the condition of the oil as the dielectric value is assumed to remain constant. In addition, this is not in line with the A.S.T.M. methods of testing power factor and dielectric value as they are tested at only one temperature. Item 5 does not agree with the steam immersion studies because the amount of water an oil will tolerate has little significance as far as transformers are concerned.

One would assume from this paper that the presence of water in insulating oil is the only condition that affects the power factor and dielectric loss. This is not true, inasmuch as dissolved varnish, carbon particles, soaps, etc., have a decided effect on these tests. Similarly there is no proof that the Doble method is measuring only water. Low molecular weight acids, hydroxy acids, and other impurities are present in the distillate from the oil and it is questionable that a carbon filter will selectively absorb only the impurities.

MR. FRANK C. DOBLE (*author*).—The comments by Messrs. Snyder and Sloat indicate that it would be helpful to some readers of this paper if references were furnished to substantiate certain statements made by the author in the brief treatment of the significance of water in oil. One set of answers will be given since several of the comments cover common ground.

Substantiation of the author's statement ". . . it is well known that under certain conditions as little as 10 to 20 ppm. of water in oil will greatly reduce its dielectric-breakdown value and constitute an immediate hazard . . (yet) under other conditions, several hundred parts per million of water may be present in an insulating oil without significantly reducing its electrical insulating value . . ." may be found in the comprehensive treatment by F. M. Clark, cited as footnote 3 in the Doble paper, and also in the "Standard Handbook for Electrical Engineers" (McGraw Hill), and in an article by Colby W. Bryden.[4]

A full explanation of the author's use of the terms "low-loss" and "high-loss" water to describe certain states or conditions of water in insulating oil is given in a paper presented by the author before the National Research Council at Harvard College, Nov. 2, 1939, and in the author's discussion[5] of F. M. Clark's paper.

It appears that three questions have been raised concerning the author's statements, "Although the line of demarcation between high-loss and low-loss water varies with conditions, such as temperature, it is possible to set up a readily reproducible value as a standard for a given condition. For this purpose, a dielectric-loss or power factor measurement can be used as an index to differentiate between high-loss and low-loss water with a base line selected at will to meet a desired condition."

1. Of what significance is a base line which can be arbitrarily selected at will?

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[4] Colby W. Bryden, "The Deterioration and Maintenance of Oil," *Iron and Steel Engineer*, February, 1939, p. 274.

[5] F. M. Clark, "Water Solution in High-Voltage Dielectric Liquids," *Transactions*, Am. Inst. Electrical Engrs., Vol. 59, p. 1193 (1940). F. C. Doble commentary on this paper.

In a laboratory study of new oils where tolerances are close, an arbitrarily selected value of, say, 0.02 per cent power factor may be used as a line of demarcation between high-loss and low-loss water, and this same line can be used subsequently in continued or similar studies. In field work, where oil power factors are usually much above 0.02 per cent, and working margins are wider, partly because of contaminants, the selection of 2 per cent power factor might be more feasible. Thus in one case the arbitrary value of 0.02 per cent power factor is significant and in another case a power factor value of 2 per cent may be equally valuable. An analogy, using the terms *hot* and *cold*, may serve to clarify the variable base-line problem. For example, a person may select 70 F. as the optimum temperature of a living room, considering higher temperatures too hot and lower temperatures too cool. However, the same individual would be likely to select 40 F. as the optimum temperature for his refrigerator and hold that temperatures above 40 F. were too hot and lower temperatures too cold. Obviously, the selection of more than one base line is significant.

2. How can power factor, which responds to impurities in oil other than water be used to differentiate between two water-oil conditions?

In a condition where water is present in an oil which also contains other contaminants, a low power factor value will prove that whatever water is present is in a low-loss form.

The presence of high-loss water in such an oil will be indicated by a high power factor reading. However, when other contaminants are known or suspected to be present, further tests to differentiate the loss due to water from the losses caused by other contaminants must be made.

3. How can high-loss and low-loss water be differentiated quantitatively?

A quantitative differentiation between high-loss and low-loss water can easily be made if desired. From a sample rated as containing high-loss water, a cut is analyzed for the total water content. Another cut from the same sample is centrifuged to remove the high-loss water, and the remainder is analyzed for its total water content which will represent the amount of low-loss water involved. The difference between these two water contents shows the amount of high-loss water present.

ELECTRICAL QUALITY CLASSIFICATION OF RAW MICA BY A RAPID, DIRECT-READING TEST METHOD*

By Kenneth G. Coutlée[1]

Synopsis

The rapid direct-reading test method for classifying dielectric loss of raw mica 0.010 to 0.030 in. thick in terms of Q at 1 megacycle, specified in A.S.T.M. Tentative Specifications for Natural Block Mica and Mica Films Suitable for Use in Fixed Mica-Dielectric Capacitors (D 748 – 45 T)[2] has been the subject of a round-robin test by Subcommittee IX on Mica Products of A.S.T.M. Committee D-9 on Electrical Insulating Materials. Tests were made by five consumers and two producers of mica on samples of electrically selected low visual quality block mica of 14 different types and various colors from world-wide sources.

All participants were found to be in close agreement using the rapid test method, and the Q calibration of the various meter ranges as determined by Q measurements of the test specimens by conventional test methods in accordance with A.S.T.M. Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (D 150 – 45 T)[3] was found to be satisfactory for electrical quality classification of mica.

The wartime need for a quick test to evaluate the electrical quality of raw mica used in huge quantities in electronic devices for the armed forces was fulfilled by the Rapid Direct Reading Method incorporated in A.S.T.M. Tentative Specifications D 748 – 45 T.[2] Subcommittee IX on Mica Products of A.S.T.M. Committee D-9 on Electrical Insulating Materials recently conducted a round-robin test of dielectric loss of raw mica in block form in terms of Q at 1 megacycle using this method. Other methods of measuring Q and dielectric constant specified in A.S.T.M. Tentative Methods D 150 – 45 T[3] also were employed by some participants for comparison purposes. The purpose of the round-robin test was threefold: (1) to determine the degree of agreement between various laboratories using the rapid method; (2) to check the Q calibration of the meter used in the rapid test method; and (3) to determine whether circular lead foil electrodes 2 in. in diameter, affixed with petrolatum, were satisfactory for measuring the Q and dielectric constant of block mica approximately 0.020 in. thick, at 1 megacycle. A total of seven laboratories participated in the round-robin test which included five consumers (R.C.A. Victor Division of the Radio Corporation of America, Western Electric Co., Bureau of Ships, Signal Corps, and Bell Telephone Laboratories, Inc.) and two producers of mica (Ford Radio and Mica

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* Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] Member of Technical Staff, Bell Telephone Laboratories, Inc., New York, N. Y.

[2] 1945 Supplement to Book of A.S.T.M. Standards, Part III, p. 322.

[3] *Ibid.*, p. 275.

Corp. and Mica Insulator Co.). An analysis of the test data received from these seven laboratories is given in the following and shows that all of the objectives of the test were satisfactorily met.

HISTORY OF RAPID TEST METHOD

It might be well at the outset to review briefly the events leading up to the development of electrical methods of quality control of mica and their incorporation in A.S.T.M. Tentative Specifications D 748. The critical wartime shortage of high-quality mica resulting from an unprecedented demand for mica capacitors was greatly aggravated in the early period of the war by the sharp decrease in the supply from India, at that time the chief source of electrical mica.

The War Production Board took steps to insure that the supply from established sources would be increased; develop new sources in the United States, South and Central America, and Canada; and inaugurate a mica conservation program. One phase of the Government mica program was concerned with the development of positive electrical tests that could be used at mine locations for rapidly classifying the electrical quality of mica for capacitor use (Fig. 1) to supplement visual quality classification in A.S.T.M. Methods D 351, and was carried out by Bell Telephone Laboratories, Inc., in cooperation with the National Research Council. This mica research project[4, 5] ultimately culminated in the development of two portable, battery-operated electrical test methods, one for classifying Q at 1 megacycle of block mica or mica splittings and the other a method for locating conducting inclusions and mechanical faults that might adversely influence the dielectric strength of block mica or splittings.

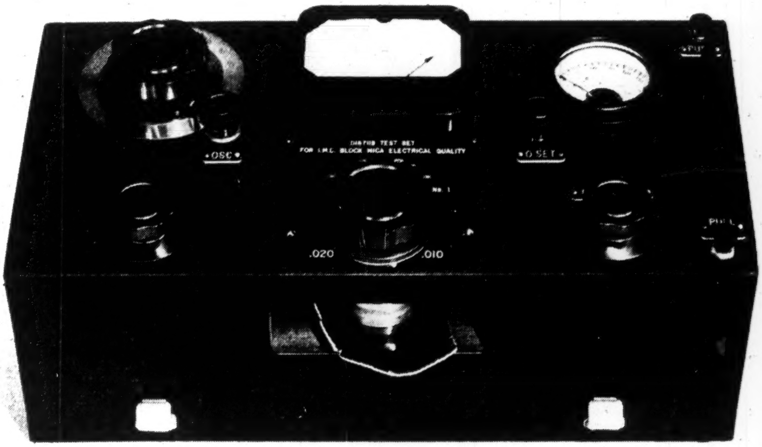

FIG. 1.—The Mica Q Test Set.

The experimental results of this mica research project were revealed to members of Section 3 of Subcommittee IX of Committee D-9 on May 12, 1943, and copies of all reports published in connection with this project were sent to each member. As a result of this close cooperation between the National Research Council Mica Project Committee

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[4] K. G. Coutlée, "Saving Mica by Testing," *Bell Laboratories Record*, September, 1944.

[5] K. G. Coutlée, "Judging Mica Quality Electrically," *Transactions*, Am. Inst. Electrical Engrs., Vol. 64, p. 735 (1945).

TABLE I.—A.S.T.M. ROUND-ROBIN *Q* TESTS OF BLOCK MICA AT 1 MEGACYCLE EMPLOYING THE RAPID METHOD AND OTHER METHODS SPECIFIED IN A.S.T.M. SPECIFICATIONS D 748.

Block Mica	Sample	Thickness, in.	Rapid Method *Q* Classification (A.S.T.M. Specifications D 748)														*Q* Value and Dielectric Constant (A.S.T.M. Method D 150)									
			Bell Telephone Labs., Inc.		Western Electric Co.		Bureau Ships		Signal Corps		Ford Radio and Mica Corp.		Radio Corp. America		Mica Insulator Co.		*Q* Value					Dielectric Constant				
																	Bell Telephone Labs., Inc.	Western Electric Co.	Bureau Ships	Signal Corps	Radio Corp. America	Bell Telephone Labs., Inc.	Western Electric Co.	Bureau Ships	Signal Corps	**Radio Corp America**
			Q Range	Meter Reading	*Q* Range	Meter Reading	*Q* Range	Meter Reading	*Q* Range	Meter Reading	*Q* Range	Meter Reading	*Q* Range	Meter Reading	*Q* Range	Meter Reading	Bridge	Bridge	*Q* Meter^b	*Q* Meter^b	Bridge	Bridge	Bridge	*Q* Meter^b	*Q* Meter^b	**Bridge**
Lot A—India (Ruby) **Stained** **Class C-1 BB^a**	No. 1	0.0186	E-1	97	E-1	97	E-1	97	E-1	97+	E-1	96	E-1	95+	E-1	96	4600	5260	4720	2 900	3670	6.58	6.63	6.71	6.61	**6.79**
	No. 6	0.0189	E-1	98+	E-1	98	E-1	98	E-1	98+	E-1	98	E-1	98+	E-1	98	4300	4320	3480	5 430	3790	6.97	6.97	7.03	7.03	**6.97**
	No. 7	0.0205	E-1	99	E-1	98	E-1	98	E-1	99	E-1	98+	E-1	99	E-1	99	3900	4030	4000	5 430	3380	6.87	6.87	6.90	6.90	**6.90**
	No. 8	0.0199	E-1	98	E-1	98	E-1	98	E-1	99	E-1	99	E-1	99+	E-1	98+	5200	6250	7130	11 230	4420	6.74	6.74	6.77	6.79	**6.74**
	No. 9	0.0185	E-1	98	E-1	97	E-1	97	E-1	98	E-1	98	E-1	97	E-1	99	4000	3300	4440	5 620	2920	5.79	5.76	5.68	6.44	**5.65**
Lot B—Brazil (Ruby) **Stained B** **Class C-1 CB^a**	No. 7	0.0174	E-1	97	E-1	98	E-1	97	E-1	97+	E-1	96+	E-1	97	E-1	97	4800	4460	3350	6 500	3600	6.71	6.63	6.69	6.99	**6.67**
	No. 8	0.0192	E-1	95	E-1	96	E-1	96	E-1	96+	E-1	96+	E-1	95+	E-1	96	1700	1810	1700	1 320	1700	5.27	5.69	5.36	6.01	**5.22**
	No. 9	0.0204	E-1	98	E-1	98	E-1	98	E-1	98	E-1	97	E-1	97+	E-1	98	3500	3510	3300	4 030	3360	6.65	6.65	6.67	6.71	**6.67**
	No. 10	0.0200	E-1	97	E-1	98	E-1	97	E-1	97	E-1	96	E-1	95	E-1	97	3500	2770	3150	2 970	2910	5.44	5.50	5.30	5.47	**5.25**
	No. 11	0.0183	E-1	97	E-1	98	E-1	97	E-1	97+	E-1	97	E-1	98	E-1	99	4000	3890	3940	3 710	4200	6.59	6.59	6.61	6.87	**6.59**
Lot D—S.D. (Ruby) **2nd Quality** **Class C-1 C + B^a**	No. 7	0.0178	E-1	98	E-1	98	E-1	98	E-1	99	E-1	97	E-1	98	E-1	98+	2700	2670	2560	3 080	2490	6.65	6.65	6.71	6.68	**6.68**
	No. 8	0.0189	E-1	97	E-1	98	E-1	97	E-1	98+	E-1	97	E-1	97+	E-1	97+	3400	3040	3600	2 540	3460	5.73	5.70	5.73	5.86	**5.75**
	No. 9	0.0185	E-1	97	E-1	98	E-1	96	E-1	96+	E-1	96	E-1	96+	E-1	97	2600	2850	2430	2 610	2620	5.92	6.07	6.00	6.10	**6.05**
	No. 10	0.0187	E-1	97	E-1	98	E-1	97	E-1	97+	E-1	97	E-1	97+	E-1	98	3300	3840	2820	3 990	3430	6.80	6.80	6.86	6.83	**6.80**
	No. 11	0.0190	E-1	95	E-1	96	E-1	95	E-1	95	E-1	95	E-1	95	E-1	96	2200	2150	2170	3 210	1970	5.43	5.46	5.41	5.99	**5.38**
Lot E—N.C. (Green) **2nd Quality** **Class C-1 AB^a**	No. 2	0.0191	E-1	98	E-1	98	E-1	97	E-1	97+	E-1	98	E-1	97	E-1	98	5100	5520	4560	8 650	3790	6.58	6.75	6.69	6.50	**6.66**
	No. 7	0.0185	E-1	97	E-1	98	E-1	97	E 1	97+	E-1	97	E-1	97	E-1	98	2800	3330	3290	5 280	3380	6.94	6.94	6.99	6.99	**6.96**
	No. 8	0.0194	E-1	98	E-1	99	E-1	97	E-1	98+	E-1	98	E-1	98	E-1	99	3600	3900	3340	5 450	3830	6.85	6.85	6.90	6.87	**6.87**
	No. 9	0.0203	E-1	98	E-1	99	E-1	98	E-1	99	E-1	98+	E-1	98	E-1	99+	4200	3470	2820	4 830	3170	6.99	6.99	7.01	7.01	**7.01**
	No. 10	0.0200	E-1	97	E-1	98	E-1	96	E-1	97	E-1	97	E-1	97	E-1	97+	3500	3400	3180	3 250	2930	6.80	6.80	6.83	6.83	**6.80**
Lot F—N.C. (Ruby) **2nd Quality "Spotted"** **Class C-1 BB^a**	No. 2	0.0202	E-1	100	E-1	99	E-1	99	E-1	97+	E-1	99	E-1	99	E-1	100	4500	4560	4450	7 470	4590	6.86	6.86	6.89	6.89	**6.86**
	No. 6	0.0189	E-1	95	E-1	96	E-1	95	E-2	93	E-1	95	E-1	95	E-1	96	2700	2320	2460	2 670	2335	6.10	6.05	6.07	6.37	**6.02**
	No. 7	0.0196	E-1	98+	E-1	98	E-1	98	E-1	96+	E-1	98+	E-1	98	E-1	99	3400	4170	4150	5 640	3570	6.79	6.79	6.84	6.81	**6.81**
	No. 8	0.0189	E-2	93	E-2	93	E-2	93	E-2	92	E-2	93	E-2	93	E-2	93+	2900	3030	2580	3 670	2950	6.63	6.71	5.79	5.79	**6.85**
	No. 9	0.0201	E-1	96	E-1	97	E-1	95	E-2	93	E-1	96+	E-1	95+	E-1	96	2800	2520	3000	2 730	2395	6.85	6.87	6.90	6.87	**6.87**
Lot H—N.H. (Ruby) **2nd Quality** **Class C-1 BC^a**	No. 1	0.0190	E-1	98	F-1	99	E-1	98	E-1	98	E-1	98	E-1	97+	E-1	98+	2800	3000	3080	3 610	2800	6.69	6.69	6.73	6.73	**6.64**
	No. 2	0.0190	E-1	97	E-1	97	E-1	97	E-1	97	E-1	96+	E-1	96+	E-1	97	3300	3140	3020	2 740	2990	6.59	6.59	6.59	6.56	**6.56**
	No. 4	0.0187	E-1	97	E-1	97	E-1	97	E-1	97	E-1	96	E-2	94+	E-1	96+	2100	2140	2060	2 540	1927	6.14	6.21	6.27	6.30	**6.35**
	No. 5	0.0181	E-1	97	E-1	97	E-1	97	E-1	97+	E-1	97	E-1	96+	E-1	97	3700	4800	3800	4 130	2815	6.16	6.24	6.31	6.31	**6.37**
	No. 7	0.0202	E-1	98	E-1	99	E-1	98	E-1	99+	E-1	98	E-1	98+	E-1	99	5900	4760	4840	10 100	4380	6.75	6.71	6.77	6.80	**6.77**
Lot I—N.C. (Green) **2nd Quality "Spotted"** **Class C-1 BC^a**	No. 7	0.0198	E-1	97	E-1	98	E-1	97	E-1	97	E-1	98	E-1	96+	E-1	98	4300	4870	4450	6 520	4080	6.78	6.72	6.78	6.72	**6.75**
	No. 8	0.0183	E-1	96	E-1	97	E-1	96	E-1	96	E-1	96	E-1	96	E-1	96+	3100	3790	2840	2 010	2320	6.67	6.71	6.79	6.81	**6.79**
	No. 9	0.0185	E-1	98	E-1	99	E-1	98	E-1	98+	E-1	98	E-1	97	E-1	99	5300	5900	4900	7 220	4900	6.89	6.89	6.91	6.94	**6.91**
	No. 10	0.0194	E-1	96	E-1	97	E-1	96	E-1	96+	E-1	96+	E-1	96	E-1	97	3700	3750	3260	3 520	3115	6.73	6.70	6.78	6.73	**6.73**
	No. 11	0.0190	E-1	98	E-1	99	E-1	98	E-1	98	E-1	97	E-1	96+	E-1	98	4200	4320	3970	5 590	3390	6.86	6.83	6.91	6.89	**6.86**
Lot J—N.C. (Ruby) **2nd Quality** **Class C-1 AB^a**	No. 1	0.0202	E-1	97	E-1	98	E-1	98	E-1	98+	E-1	98	E-1	97	E-1	98	3800	5260	4320	6 190		6.72	6.69	6.72	6.51	
	No. 4	0.0202	E-1	97	E-1	97	E-1	98	E-1	97	E-1	97	E-1	95+	E-1	97	3400	4200	3640	3 420	3750	6.63	6.69	6.77	6.77	**6.81**
	No. 7	0.0183	E-1	97	E-1	98	E-1	98	E-1	97+	E-1	97	E-1	96+	E-1	97	3000									
	No. 10	0.0191	E-1	97																						

Lot J—N.C. (Ruby) 2nd Quality Class C-1 AB[a]	No. 1	0.0202	E-1	97	E-1	98	E-1	98	E-1	98+	E-1	98	E-1	97	E-1	98	3800	5260	4320	6 190		6.72	6.69	6.72	6.51	....
	No. 4	0.0202	E-1	97	E-1	97	E-1	98	E-1	97	E-1	97	E-1	95+	E-1	97	3400	4200	3640	3 420	3750	6.63	6.69	6.77	6.77	**6.81**
	No. 7	0.0183	E-1	97	E-1	98	E-1	98	E-1	97+	E-1	97	E-1	96+	E-1	97	3000	3000	3060	3 820	....	6.81	6.81	6.87	6.87	....
	No. 10	0.0191	E-1	97	E-1	98	E-1	97	E-1	98	E-1	97	E-1	96+	E-1	97+	3700	5370	4460	5 840	....	6.39	6.43	6.51	6.50	...
Lot L—Canada (Green) 2nd Quality Class C-1 AA[a]	No. 5	0.0189	E-1	95	E-1	97	E-1	95	E-1	95	E-1	95	E-2	94	E-1	95	4800	6060	4300	3 540	2830	6.18	6.18	6.23	6.34	**6.21**
	No. 7	0.0182	E-1	97	E-1	98	E-1	97	E-1	97+	E-1	97	E-1	96+	E-1	97	2900	2750	2680	2 960		6.75	6.75	6.80	6.83	....
	No. 8	0.0183	E-2	94	E-1	98	E-1	95	E-1	95+	E-1	96	E-1	95	E-1	96	1800	2070	1870	1 740	1695	7.00	7.07	7.16	7.16	**7.19**
	No. 9	0.0187	E-1	98	E-1	99	E-1	98	E-1	98+	E-1	98	E-1	97	E-1	96	3900	3420	3680	5 900	....	6.80	6.77	6.85	6.85	....
	No. 10	0.0178	E-1	97	E-1	98	E-1	97	E-1	97+	E-1	97	E-1	96	E-1	97	3600	3050	4150	4 320	....	6.73	6.73	6.80	6.78	....
Lot M—India (Ruby) Good Stained Class C-1 BB[a]	No. 1	0.0202	E-1	97	E-1	98	E-1	99	E-1	98+	E-1	97	E-1	97+	E-1	97+	3400	....	2780	3 250	....	5.74	....	5.86	5.80	....
	No. 2	0.0183	E-1	99	E-1	99	E-1	100	E-1	99+	E-1	98+	E-1	99	E-1	98+	4900	....	4410	6 330	3520	6.63	....	6.81	6.81	**6.84**
	No. 3	0.0191	E-1	96	E-1	97	E-1	97	E-1	96+	E-1	96	E-1	96+	E-1	96	5300	....	5190	5 820	5180	6.77	....	6.85	6.88	**6.85**
	No. 4	0.0201	E-1	97+	E-1	98	E-1	98	E-1	98	E-1	97	E-1	97	E-1	97	4100	....	3460	1 190	....	6.82	....	6.90	6.90	....
	No. 5	0.0199	E-1	99	E-1	99	E-1	99	E-1	99+	E-1	98	E-1	97	E-1	98	6800	....	8070	10 900	....	6.40	....	6.54	6.57	....
Lot O—N.C. (Green) 3rd Quality Black Stained and Spotted Class C-1 BB[a]	No. 1	0.0192	E-1	97	E-1	98	E-1	98	E-1	98+	E-1	97	E-1	97+	E-1	97+	2900	4530	4030	5 540	....	6.50	6.47	6.63	6.58	....
	No. 3	0.0189	E-1	97	E-1	97	E-1	97	E-1	97+	E-1	98	E-1	95+	E-1	97	3700	3800	3280	3 230	....	6.61	6.61	6.66	6.63	....
	No. 4	0.0183	E-1	95	E-1	97	E-1	98	E-1	96+	E-1	96	E-1	95	E-1	96	2500	2700	2360	2 660	....	6.68	6.68	6.73	6.71	....
	No. 5	0.0183	E-1	96+	E-1	97	E-1	96	E-1	96	E-1	95	E-2	94	E-1	95	2700	2970	2460	2 210	2070	6.19	6.27	6.30	6.43	**6.63**
	No. 6	0.0193	E-1	96	E-1	97	E-1	96	E-1	96+	E-1	96	E-1	95+	E-1	96	2000	1940	1350	1 600	1607	6.50	6.59	6.61	6.56	**6.64**
Lot P—N.C. (Green) 3rd Quality Reeved Class C-1 BC +[a]	No. 1	0.0182	E-1	96	E-1	97	E-1	96	E-1	96+	E-1	96	E-2	94	E-1	95+	5000	6150	4800	5 400	3280	6.00	6.06	6.09	6.11	**6.09**
	No. 2	0.0169	E-1	97+	E-1	99	E-1	98	E-1	98+	E-1	97	E-1	96	E-1	97	3200	3350	3300	3 900	....	6.46	6.39	6.43	6.41	....
	No. 5	0.0204	E-1	96	E-1	98	E-1	96	E-1	96+	E-1	95	E-1	95+	E-1	96+	2900	2900	2960	3 460	....	6.91	6.91	6.97	6.94	....
	No. 6	0.0192	E-1	97	E-1	99	E-1	97	E-1	97+	E-1	96	E-1	94+	E-1	96	2900	3470	3060	4 850	3040	6.63	6.63	6.67	6.63	**6.63**
	No. 7	0.0193	E-1	98	E-1	98	E-1	98	E-1	98	E-1	97	E-1	96+	E-1	97	3500	3890	3160	3 420	....	6.89	6.94	7.00	7.00	....
Lot Q—Canada (Green) 2nd Quality Class C-2 AB[a]	No. 1	0.0192	E-2	86	E-2	88	E-2	86	E-2	88+	E-2	88	E-2	87	E-2	88+	2100	2170	2260	1 430	....	6.86	6.86	6.82	6.79	....
	No. 2	0.0201	E-2	92	E-2	93	E-2	92	E-2	92+	E-2	92	E-2	92+	E-2	92+	4400	4660	4420	5 790	....	6.76	6.73	6.79	6.79	....
	No. 3	0.0181	E-2	90	E-2	92	E-2	90	E-2	90+	E-2	91	E-2	90+	E-2	90+	1200	1250	1100	939	....	5.73	5.81	5.70	6.17	....
	No. 5	0.0185	E-2	81	E-2	85	E-2	81	E-2	80+	E-2	81	E-2	78+	E-2	81+	2900	2670	2660	2 280	2560	6.81	6.81	6.85	6.85	**6.81**
	No. 6	0.0187	E-2	94+	E-1	96	E-2	94	E-1	95	E-2	94	E-2	94+	E-1	95+	3700	4000	4750	5 870	3000	6.77	6.72	6.83	6.80	**6.75**
Lot R—Canada (Green) 3rd Quality Class C-3 AB[a]	No. 1	0.0195	E-3	73+	E-3	73	E-3	73	E-3	72+	E-3	75	E-3	72+	E-3	73	560	770	705	348	693	6.64	6.64	6.69	6.69	**6.47**
	No. 2	0.0176	E-3	47	E-3	51	E-3	49	E-3	42+	E-3	51	E-3	50	E-3	50	790	1080	830	447	....	6.72	6.72	6.75	6.75	....
	No. 3	0.0185	E-3	51	E-3	52	E-3	71	E-3	53+	E-3	54	E-3	52+	E-3	49+	1300	1620	1250	562	....	6.70	6.70	6.75	6.75	....
	No. 4	0.0198	E-3	71	E-3	77	E-3	76	E-3	74+	E-3	76+	E-3	75	E-3	75+	1200	1260	1090	797	....	6.78	6.78	6.83	6.83	....
	No. 5	0.0193	E-3	44	E-3	53	E-3	41	E-3	47+	E-3	50+	E-3	50+	E-3	51	1500	1630	1460	1 480	1250	6.83	6.83	6.86	6.89	**6.89**

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[a] A.S.T.M. Tentative Specifications for Natural Block Mica and Mica Films Suitable for Use in Fixed Mica-Dielectric Capacitors (D 748 – 45 T), 1945 Supplement to Book of A.S.T.M. Standards, **Part III**, p. 322.

[b] Boonton 160 A Type.

Notes

1.—*Rapid method Q range for block mica 0.020-in. thick*

Range	E-1	E-2	E-3
Q	2500 min.	350 min.	50 min.
Meter reading	95-100	77-95	32-77

2.—All mica samples originally were selected (unbaked) as E-1, E-2, or E-3 as the case may be, then lead foil electrodes were applied and above measurements made.

3.—Samples transported in air-tight container.

4.—*Test Conditions:*

Bell Telephone Labs.	50% RH 72 F.
Western Electric Co.	38% RH 72 F.
Bureau Ships	50% RH 75 F.
Signal Corps	50% RH 75 F
Ford Radio and Mica Corp.	>50% RH 85 F
Radio Corp. America (Test Set)	50% RH 73 F.
Radio Corp. America (Bridge)	75% RH 75 F
Mica Insulator Co.	<50% RH 75 F.

and A.S.T.M. Subcommittee IX on Mica Products it was possible to prepare and issue A.S.T.M. Specifications D 748 incorporating these test methods, by November, 1943, in time for wartime use by industry. The results of this investigation have shown that block mica can be selected electrically by test methods that are fast and reliable, for use in making highest quality as well as less critical types of capacitors and that much mica previously judged as unsuitable for such use by visual standards can be used. This effectively has increased the mica supply for capacitors by as much as 60 per cent.

Although the test methods contained in Specifications D 748 were the subject of an extensive commercial trial as part of the research program for the War Production Board it was not possible, due to the war urgency, to carry out the usual A.S.T.M. round-robin tests of the test methods prior to their inclusion in the specifications. Therefore, as soon as time permitted, a round-robin test of the rapid method of Q classification, which is considered the more critical of the two electrical test methods that were developed, was started and the results obtained are given in this paper.

TEST SPECIMENS

The test specimens used in the round-robin tests were selected by Bell Telephone Laboratories and corresponded to E-1, E-2, and E-3 electrical quality in Specifications D 748. In these three electrical-quality classification groups, E-1 is the range of lowest dielectric loss perpendicular to the plane of basal cleavage obtainable for any mica, E-2 an intermediate loss range, and E-3 a higher loss range, but still perfectly satisfactory for less critical types of capacitors which constitute a very large percentage of the total. Five representative specimens of each of 14 different varieties of ruby and green types of muscovite mica from Brazil, India, Canada, and the United States were selected, and with the exception of one lot (lot *M* India ruby "good stained") which served as a control in the tests that were made for the War Production Board all 13 others were below visual standards considered suitable for capacitor use. Detailed information concerning source, color, commercial quality and electrical quality in accordance with A.S.T.M. Specifications D 748 classification is given in Table I. Lots A, B, D, E, F, H, I, J, L, M, O, and P were selected as E-1 (Q 2500 minimum); lot Q as E-2 (Q 350 minimum); and R as E-3 (Q 50 minimum); in the manner specified in the rapid method in D 748. In other words each sample of block mica, which was approximately 0.020 in. thick, first was clamped between the 2-in. diameter test electrodes (without foil electrodes) and the Q classification as well as the reading of the meter observed. These specimens were selected from lots of mica which had been stored for a period of several months in an air-conditioned room operating at 50 per cent relative humidity, 73 F., and were not baked prior to selection.

As the round-robin test also entailed Q and dielectric constant measurements by other test methods which would require the use of foil electrodes, such electrodes were applied after the initial selection test was made. While it would have been possible for each testing laboratory to make rapid tests without electrodes and then apply them for conventional tests, it was felt too many variables might be introduced into the test if such a procedure was followed. Lead-foil electrodes 0.0005 in. thick and 2 in. in diameter were mounted on both faces of each specimen using a minimum quantity of U.S.P. amber petrolatum as an adhesive. Since the mica specimens were transparent, this permitted accurate alignment of the electrodes. Visible contamination and finger marks on the mica specimens were removed by rubbing with a soft lint-free cotton cloth prior to the application of electrodes.

At the completion of the round-robin tests, the lead-foil electrodes were removed and the samples immediately retested by the rapid method, care being exercised to have the test electrodes contact the same area originally covered by the foil electrodes. The values obtained are shown in Table II and were either the same or generally within one scale division of those obtained with foil electrodes on the samples.

It is interesting to note that the foil electrodes remained in firm contact with the mica all through the round-robin tests. In fact the adherence was so good that some difficulty was experienced in removing them. No formation of gas bubbles under the foils of any of the samples was observed such as usually occurs when left in contact with plastic specimens for a long period of time. This seems to indicate that if any gas was evolved from the specimens perpendicular to the plane of basal cleavage, especially those containing excessive amounts of air bubbles, such as lots A, B, and D, it was not in sufficient quantity to form pockets under the foils.

TABLE II.—RAPID METHOD *Q* CLASSIFICATION OF BLOCK MICA USED IN A.S.T.M. ROUND-ROBIN *Q* TESTS IMMEDIATELY AFTER REMOVAL OF FOIL ELECTRODES.

Lot	Sample	*Q* Range	Meter Reading	Air Pockets[a]
A	No. 1	E-1	96+	Yes
	No. 6	E-1	98+	Yes
	No. 7	E-1	99+	No
	No. 8	E-1	98+	No
	No. 9	E-1	98+	No
B	No. 7	E-1	97+	Yes
	No. 8	E-1	95+	No
	No. 9	E-1	98	No
	No. 10	E-1	96	No
	No. 11	E-1	98	No
D	No. 7	E-1	98	No
	No. 8	E-1	97+	No
	No. 9	E-1	96+	No
	No. 10	E-1	97	No
	No. 11	E-1	95+	No
E	No. 2	E-1	96+	Yes
	No. 7	E-1	97+	No
	No. 8	E-1	97+	No
	No. 9	E-1	99	No
	No. 10	E-1	97	Yes
F	No. 2	E-1	99+	Yes
	No. 6	E-1	95	Yes
	No. 7	E-1	98	Yes
	No. 8	E-2	94+	Yes
	No. 9	E-1	95	No
H	No. 1	E-1	98	Yes
	No. 2	E-1	96+	Yes
	No. 4	E-1	96	Yes
	No. 5	E-1	96+	Yes
	No. 7	E-1	98+	Yes
I	No. 7	E-1	97	No
	No. 8	E-1	95	No
	No. 9	E-1	98	No
	No. 10	E-1	96	No
	No. 11	E-1	97	No
J	No. 1	E-1	97	No
	No. 4	E-1	96+	No
	No. 7	E-1	97	No
	No. 10	E-1	95+	Yes
L	No. 5	E-1	95	Yes
	No. 7	E-1	97+	No
	No. 8	E-1	95	No
	No. 9	E-1	99	No
	No. 10	E-1	96	No
M	No. 1	E-1	97	No
	No. 2	E-1	99	No
	No. 3	E-1	96+	No
	No. 4	E-1	97	Yes
	No. 5	E-1	99	Yes
O	No. 1	E-1	98+	Yes
	No. 3	E-1	97	Yes
	No. 4	E-1	96+	Yes
	No. 5	E-1	95+	Yes
	No. 6	E-1	97	Yes
P	No. 1	E-1	96	Yes
	No. 2	E-1	97+	Yes
	No. 5	E-1	96+	Yes
	No. 6	E-1	97	Yes
	No. 7	E-1	97+	Yes
Q	No. 1	E-2	87	No
	No. 2	E-2	91+	Yes
	No. 3	E-2	91	Yes
	No. 5	E-2	85	No
	No. 6	E-1	95	No
R	No. 1	E-3	77+	No
	No. 2	E-3	57+	No
	No. 3	E-3	67+	No
	No. 4	E-3	72+	No
	No. 5	E-3	41+	No

[a] Air pockets due to partial delamination which extended into the region covered by the foil electrodes.

TEST PROCEDURE

The mica test specimens with foil electrodes attached were circulated among the various testing laboratories being transported in an air-tight container. Each sample was enclosed in a cellophane envelope marked with the sample designation. The sample designation also was scratched into one face of each test specimen. At the start of a test all the specimens were removed from the container and then taken from the cellophane envelope one at a time and tested. So as to reduce variations to a minimum, instructions were given to connect the foil electrode on the designation side of the sample to the ungrounded side of whatever test circuit was being used. This was done to eliminate possible variations as it had been observed, particularly in the case of high-loss samples, that oddly enough the power factor was sometimes influenced by which side of the specimen was connected to ground.

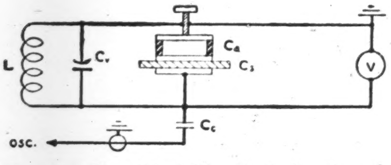

FIG. 2.—Simplified Circuit Indicating the Method of Testing for *Q* or Power Factor.

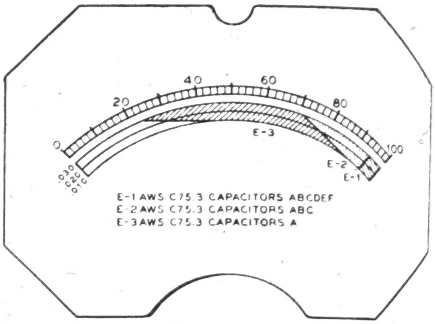
0
20
40
60
80
100
E-3
E-2
E-1
.030
.020
.010
E-1 AWS C75.3 CAPACITORS ABCDEF
E-2 AWS C75.3 CAPACITORS ABC
E-3 AWS C75.3 CAPACITORS A

FIG. 4.—Meter Scale of the Mica *Q* Test Set.

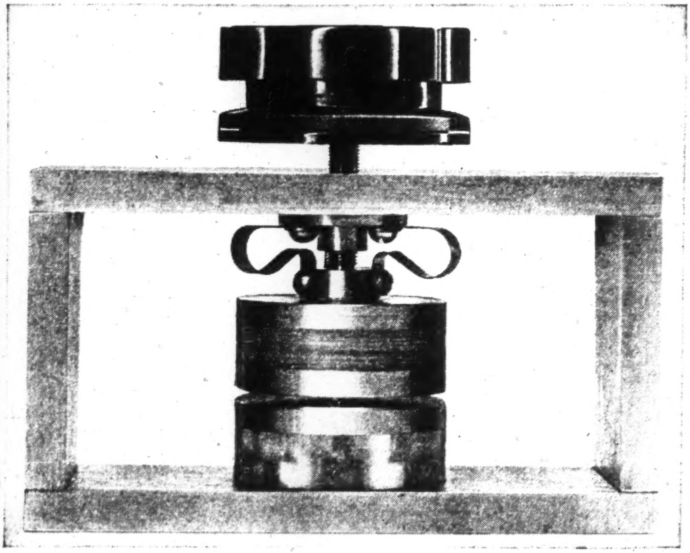

FIG. 3.—Test Electrodes.

In the rapid method tests (Fig. 2) the sample was located so that the foil electrodes registered accurately with respect to the test set electrodes (Fig. 3) and clamped in that position. The *Q* balance dial of the test circuit was adjusted to give maximum deflection of the meter and the *Q* range and meter reading (0 to 100 scale) obtained were recorded and this procedure was repeated for each sample. It is not necessary to read the meter scale (Fig. 4) in the normal use of this test method but merely observe the *Q* range. However, the scale reading also was required in the round-robin tests in order to permit a comparison of individual test sets.

During measurements of Q and dielectric constant by conventional test methods in A.S.T.M. Methods D 150 a somewhat similar procedure to that used in the rapid test method was followed. However, in this test the sample was not clamped under pressure between electrodes as it was in the rapid test method. Rather, contact to the foil electrode on the ungrounded side of the circuit was made by a gold-plated saw-tooth piece of brass about $\frac{3}{8}$ in. wide soldered to a short length of No. 14 B. & S. gage bare copper wire. Connection to the lower grounded electrode was made by laying the sample on a gold-plated brass disk about $1\frac{1}{4}$ in. in diameter. The zero reading of the circuit was taken with a $\frac{1}{8}$-in. air gap between the upper contact and the foil electrode. During measurement, the No. 14 wire was bent so as to provide a definite pressure between the saw-tooth contact and the foil electrode. The test methods employed by the various testing laboratories collaborating in these tests are as follows:

BELL TELEPHONE LABORATORIES, INC.

Rapid Method Tests:

B.T.L. D-167113 mica Q test set.

Q and Dielectric Constant Tests:

B.T.L. precision type capacitance and conductance bridge in conjunction with an oscillator and detector amplifier with a visual balance indicator (meter).

WESTERN ELECTRIC CO.

Rapid Method Tests:

Boonton 130A mica Q test set.

Q and Dielectric Constant Tests:

Precision type capacitance and conductance bridge similar in design to that used by B.T.L.

BUREAU OF SHIPS

Rapid Method Tests:

Boonton 130A mica Q test set.

Q and Dielectric Constant Tests:

Boonton 160A Q meter (parallel method).

SIGNAL CORPS

Rapid Method Tests:

Boonton 130A mica Q test set.

Q and Dielectric Constant Tests:

Boonton 160A Q meter (parallel method).

FORD RADIO AND MICA CORP.

Rapid Method Tests:

Boonton 130A mica Q test set.

Q and Dielectric Constant Tests:

None made.

RADIO CORPORATION OF AMERICA

Rapid Method Tests:

Boonton 130A mica Q test set.

Q and Dielectric Constant Tests:

Precision type capacitance and conductance bridge of modified Leeds and Northrup design in conjunction with oscillator, detector amplifier, and earphones for obtaining balance.

MICA INSULATOR CO.

Rapid Method Tests:

Boonton 130 mica Q test set.

Q and Dielectric Constant Tests:

None made.

The results of the above tests are recorded in Table I.

DISCUSSION OF RESULTS

Rapid Method Tests:

The agreement among the various laboratories on the Q classification of the block mica samples by the rapid method was found to be quite close. Test results on the 59 E-1 samples showed that four laboratories found them to be 100 per cent within test set limits and the other three that the classification was 98, 97, and 93 per cent correct. One sample was excluded from the E-1 group as all participants reported it as E-2. In the final tests when the foil electrodes had been removed this sample proved to be E-2 indicating that it initially was misclassified. The deviation from the mean test set value of any one E-1 sample was one scale division or less in the majority of cases and two divisions maximum in only a few instances. Only five E-2 samples were included in the test and four laboratories reported them 100 per cent within test-set limits, the three remaining laboratories finding them 80 per cent within limits. However, since all of the latter three found that the nonconforming specimen met E-1 limits, the number of specimens actually meeting minimum E-2 test-set limits was 100 per cent. All participants found the five E-3 samples to be 100 per cent within test-set limits.

This rather excellent agreement among the various rapid method test sets is even more striking when one takes into consideration the fact that in the majority of cases it was the operators first experience with the test equipment when the round-robin samples were run. This emphasizes the simplicity and reliability of the test method.

Q and Dielectric Constant Tests:

The Q tests on the E-1 samples showed that agreement with Q limits for E-1 block mica reported by the testing laboratories was over a range of 91 to 96 per cent correct. This included some borderline samples with Q values within 15 per cent of the minimum Q limit of 2500 for E-1 block mica. This is the estimated accuracy of a bridge measurement of a Q of this order of magnitude, at 1 megacycle. Considering the high order of Q being measured, the agreement among the five laboratories making tests on individual E-1 specimens is considered quite good in the majority of cases and affords an opportunity to compare bridge and resonant circuit Q meter test methods.

In a few cases the Q value of some E-1 samples exceeded minimum limits even though they were within limits by the rapid method test. Likewise, some rapid method E-2 samples were found to have Q values that met E-1 Q limits by conventional test methods.

Some studies were carried out in an effort to determine the reasons for such behavior. The effect of pressure was first investigated since in the rapid tests pressure is present which does not exist in the regular Q tests. The test electrodes were borrowed from the rapid method and connected to a bridge. The 20-mmf. fixed-air capacitor built into the upper movable electrode of the test electrodes was shorted out so that the electrodes on either side of the sample merely served as contacts to the foil electrodes. Under a pressure normally applied, the Q values were found to be in close agreement with those obtained without pressure. Q measurements next were made under the same setup except that the sample was placed in series with the 20-mmf. fixed-air capacitor thereby simulating the rapid test method condition. The Q values obtained in this manner on those E-2 samples that met E-1 Q limits were found to be lower and more nearly in line with the rapid method E-2 classification. Similarly, tests on E-3 samples also gave lower Q values than obtained by standard tests. Such effects seem to be peculiar to mica and were not found in such materials as hard rubber, phenol fiber, vinylite and alsifilm of equivalent thickness under simliar conditions of test.

The five testing laboratories that made dielectric constant tests furnished capacitance values for E-1, E-2, and E-3 samples. Dielectric constant values were calculated by Bell Telephone Laboratories using specimen thickness and foil electrode area values obtained by them. Very good agreement on any one specimen in the majority of cases is evident from the test results. There was no significant difference in dielectric constant at 1 megacycle between the three electrical qualities of lots L(E-1), Q(E-2), and R(E-3) mica all of which were green Canadian from the same mine.

CONCLUSIONS

Based on the test results obtained by seven testing laboratories on samples of block mica of 14 varieties of ruby and green types of muscovite from world-wide sources it is concluded that:

1. Very close and satisfactory agreement was found between all seven testing laboratories using the rapid, direct-reading test method in A.S.T.M. Specifications D 748.

2. The *Q* calibration of the three *Q* ranges of the rapid, direct-reading test method as determined by *Q* tests by other methods of test in A.S.T.M. D 150 is satisfactory for the purpose of electrical quality classification of mica used in capacitors.

3. The dielectric constant at 1 megacycle of the wide variety of types and qualities of muscovite mica included in the group of test samples is quite uniform.

4. A greater dielectric loss of some samples of block mica is sometimes indicated by the series circuit of the rapid test method than is obtained by regular test methods. Such peculiar behavior seems to be characteristic of some mica and not observed in hard rubber, phenol fiber, vinylite, and alsifilm under identical test conditions. In view of this, the rapid test method would appear to be somewhat more discriminating than normal test methods, but not unduly so, as borne out by the high yield of E-1 mica from all visual qualities of mica from world-wide sources as shown by recently published data. Such discrimination, however, serves as an added factor of safety.

5. Foil electrodes 0.0005 in. thick and 2 in. in diameter in conjunction with petrolatum as an adhesive are suitable for measuring *Q* and dielectric constant of block mica approximately 0.020 in. thick by test methods specified in A.S.-T.M. Methods D 150.

DISCUSSION

Mr. S. B. Ashkinazy.[1]—On the basis of the illustrations shown in this paper, we have seen that several different pieces of mica, looking alike, have actually different electrical characteristics, which is really the ultimate in the application of this particular material. In view of this, I should like to have Mr. Coutlée's opinion as to just what is in the future in regard to the use of this test procedure. I understand there is some reluctance to supply mica under this test for electrical characteristics rather than visual standards, and not much is being purchased on this basis.

Mr. Kenneth G. Coutlée (*author's closure*).—There has been some reluctance indicated in the use of this method by some mica producers. The mica business has been carried on more or less on an expert opinion basis and before the war we were able to procure the type of mica required by having mica experts in the consumer companies and by using India ruby-type mica which had been found to possess uniform electrical quality and could be judged by visual standards such as set forth in A.S.T.M. Standard Methods of Test for Grading and Classification of Natural Mica (D 351).[2] Mica is a strategic mineral that was in critical short supply all during the war. In attempting to evaluate mica from new sources in the United States, Canada, South America and Central America by India standards it sometimes was difficult to do so, due to characteristic differences in color and appearance. In order to overcome the limitations of the visual system, electrical test methods were developed to supplement them. We always have opposition to specifications, especially from producers who do not seem particularly to like them. This is a new thing and the producers no doubt are a little bit afraid of it. They apparently are of the opinion that 100 per cent of the product must be tested, whereas we have found and shown by our tests that in the majority of cases this is not so. It is possible to sample a case of mica and come out with a fairly accurate estimate of how the mica grades up electrically. Fortunately, we found things generally better than we had expected: that is, mica is good electrically regardless of source. Even the third electrical quality—which is about equivalent to so-called low-loss phenolics in dielectric loss—could be used to make a large portion of the less critical types of mica capacitors. However, we need a definite control, so that if we do require the very best electrical type, we can select and buy it on the electrical quality classification method. If you are buying India ruby-type mica using the visual system, the chances are you will come out with a good electrical product provided you use the better visual qualities. This has been borne out by past experience and verified by recent tests. However, in the case of green-type mica, which has not been acceptable for capacitor use in the past, it now is possible to select and classify it electrically.

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[1] Materials and Process Standards Supervisor, Sperry Gyroscope Co., Inc., Great Neck, N. Y.

[2] 1944 Book of A.S.T.M. Standards, Part III, p. 504.

thereby greatly increasing the supply. Green-type mica may or may not grade up electrically as high as the ruby type, but the electrical test methods permit one to select just as good an electrical quality as the best ruby.

I am unable to predict how long the mica fabricators will adhere to their opinion with respect to the use of the electrial test in furnishing mica. I know Bell Telephone Laboratories, Inc. specifies A.S.T.M. Tentative Specifications for Natural Block Mica and Mica Films Suitable for Use in Fixed Mica-Dielectric Capacitors (D 748),[3] which incorporates the electrical tests I have discussed, on apparatus drawings where applicable. On other drawings where visual quality is desired Methods D 351[2] are specified. Subcommittee IX on Mica Products, of A.S.T.M. Committee D-9, has tried to correlate the visual and the electrical methods of classification based on the knowledge we have obtained in this study, and such information is contained in Tentative Specifications D 748.[3] This enables a mica producer to furnish the visual quality that probably will come closest to being satisfactory electrically. Of course, if he wants to be absolutely sure he is meeting this specification, he will spot check a pound or two or whatever quantity is necessary, depending upon the amount being supplied. One thing that is lacking in Tentative Specifications (D 748)[3] at the moment is a sampling method, and we are planning to include one in the near future which we hope will overcome some of the present objections.

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[3] 1945 Supplement to Book of A.S.T.M. Standards, Part III, p. 322.

DIELECTRIC STRENGTH MEASUREMENTS ON VARNISHED CAMBRIC*

An Investigation of Several Factors to Determine Their Effects on the Measured Dielectric Strength of Varnished Cambric Insulation

By A. Rufolo[1] and H. K. Graves[1]

The determination of dielectric strength of an insulating material has always been complicated by the presence of several factors known to exert considerable influence on results obtained. It has been accepted practice to control these effects as much as possible by using standard procedures. Consequently, while standardization may be reasonably successful in establishing a basis for comparison among investigators, the degree and range of effects produced by these factors may remain relatively unexplored. Using varnished cambric samples, the effects of three of these factors, namely, humidity, electrodes, and breakdown media, were studied as an approach to the general problem.

The samples obtained included four thicknesses of black varnished cambric cloth, both bias and straight-cut, representing five manufacturers. Several samples of yellow varnished cambric were also obtained. The range of the study was further restricted to include only those methods believed to be currently specified or in use in the various laboratories. The investigation was conducted with three types of electrodes, for two pre-test conditions and with breakdowns made in two media.

Certain other considerations which prompted the use of this general procedure are worthy of discussion. The 2-in. diameter electrodes were selected as being the most satisfactory for dielectric strength tests on varnished cambric cloth since they cover a relatively large area of sample and permit a reasonably large radius of curvature at the edges to minimize voltage stress concentration. The $\frac{1}{4}$-in. diameter electrodes have been used for many years in the testing of varnished cambric and possess the advantage of being equally applicable to both cloth and narrow tapes, but they have the disadvantage of higher stress concentration at the edges. The $\frac{1}{4}$ by 4-in. rectangular electrodes have also been standard for testing of other types of insulating tapes, have stress concentration comparable to that of the $\frac{1}{4}$-in. electrodes and have the advantage of being applicable to both cloth and narrow tapes, but, unlike $\frac{1}{4}$-in. electrodes, have a much higher specimen coverage. A tabulation of electrode dimensions to outline

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[1] Materials Engineer, Material Laboratory, New York Naval Shipyard, Brooklyn, N. Y.

some of the variables involved might be set up as follows:

Electrode size, in.	¼	¼ by 4	2
Shape	Circular	Rectangular	Circular
Area, sq. in.	0.049	1.0	3.14
Perimeter, in.	0.785	8.8	6.28
Radius of curvature at edges, in.	0.031	0.031	0.25

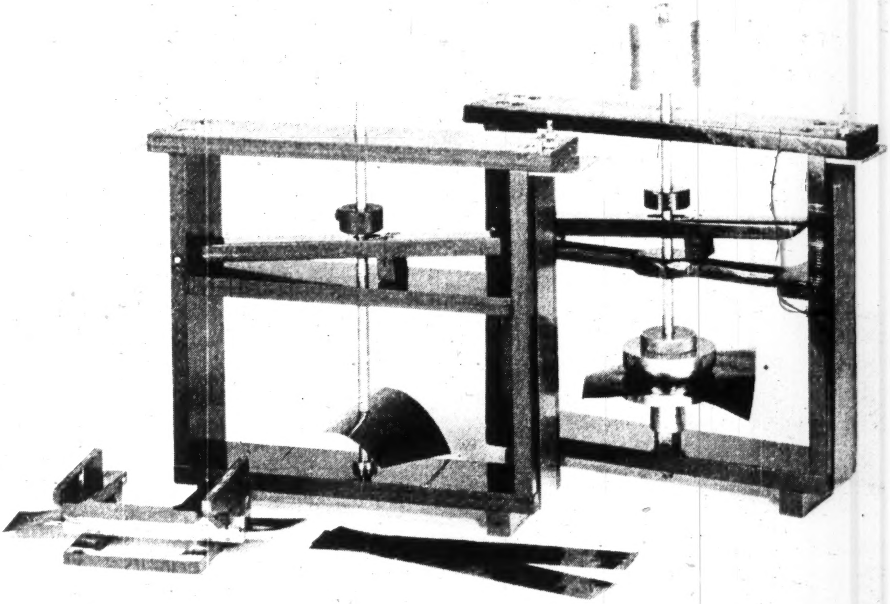


FIG. 1.—The Three Types of Electrodes Used for the Investigation.

The two conditioning humidities were chosen because they were readily attainable in any laboratory and represented extremes of low and high humidity without, however, exceeding possible service conditions.

Since there has been some controversy on the relation of dielectric breakdown measured in air to that measured with the sample immersed in transformer oil, comparative tests were run in the two media. However, only the smaller size electrodes were used to avoid the necessity of conditioning and breaking down large specimens.

TEST EQUIPMENT

A dial type micrometer, calibrated to 0.0001 in. with 2-in. diameter anvil and $\frac{1}{4}$-in. diameter presser foot under a 3-oz. dead weight, was used for all thickness measurements.

Breakdowns above 3000 v. were made on a 2-kva. transformer with a 4-coil secondary and switching arranged to give full-scale readings of 7500, 15,000, or 30,000 v. Below 3000 v. (nearly all the high humidity samples) breakdowns were made on a 3-kva. transformer with a single secondary winding. Voltmeters connected to tapped tertiary windings were used on both transformers to indicate breakdown voltages. Voltage rise in each case was manually controlled by means of variable auto-transformers.

The three types of electrodes, shown in Fig. 1, may be briefly described as follows:

(*a*) *2-in.*—Polished brass disks, 2 in. in diameter and $\frac{1}{2}$ in. thick, having edges in contact with the sample rounded to a $\frac{1}{4}$-in. radius. Faces self-aligning with 1.5-lb. pressure on specimens.

(*b*) $\frac{1}{4}$ *by 4-in.*—Polished brass bars $\frac{1}{4}$ in. wide, 4 in. long, and $\frac{1}{4}$ in. thick, with ends rounded to a $\frac{1}{8}$-in. radius parallel to the sample and with edges of contact faces rounded to a $\frac{1}{32}$-in. radius. Faces self-aligning with 1-lb. pressure on specimens.

(*c*) $\frac{1}{4}$*-in.*—Polished brass rods $\frac{1}{4}$ in. in diameter having edges in contact with specimen rounded to a $\frac{1}{32}$-in. radius. Rigidly held in alignment with 1-lb. pressure on specimens.

Standard sealed desiccators were used for conditioning of specimens. Anhydrous calcium chloride was used for the dry condition to produce a relative humidity of less than 10 per cent, and saturated solutions of potassium sulfate were used to produce the 96 per cent relative humidity. A temperature of 25 C. was maintained for the entire conditioning period.

TEST SPECIMENS

Samples used for the test were taken from rolls stored for several weeks at 25 C. and 50 per cent relative humidity. Materials were in nominal thicknesses of 5, 7, 10, and 12 mils, bias and straight-cut, black and yellow. The specimens used with 2-in. electrodes were cut 3 in. square and those used with the $\frac{1}{4}$ by 4 in. were cut into 1 by 7-in. rectangles. Specimens used with the $\frac{1}{4}$-in. electrodes were 6 in. square for tests in air and 1 by 7 in. for tests in oil.

TEST PROCEDURE

All testing was carried on under controlled room conditions of 25 C. and 50 per cent relative humidity. Complete tests were made on one sample cloth before starting another and samples awaiting test were stored in the rolls at the above conditions.

In starting a test on a sample, sufficient material was removed from the roll to provide the necessary specimens and twenty thickness measurements were made at widely distributed points on this piece prior to cutting specimens. The average of these twenty measurements was used in the calculation of dielectric strength.

Specimens were spaced $\frac{1}{2}$ in. apart on bent wire racks and placed in the conditioning desiccators. After completion of the 96-hr. conditioning period, specimens were removed from desiccators in groups of ten, stacked to minimize change in condition, and tested as rapidly as possible. Since dielectric strength involves volume rather than surface characteristics and since there was no discernible progressive change in values from the first to the last of the group of ten specimens, it was assumed that no significant gain or loss of moisture occurred between the time the sample was removed from the desiccator and the time it was tested.

Rate of rise of voltage was manually controlled at 500 v. per second with the aid of an electric stopclock connected in the transformer circuit. Twenty-five individual breakdowns were made on each cloth for each type electrode after each conditioning.

RESULTS OF INVESTIGATION

The results of test, in average volts per mil dielectric strength, for each of the 32 samples are presented graphically in Figs. 2 and 3. For interpreting the sample code, numbers represent thickness, capital letters distinguish between manufacturers, and the letters *y* and *b* indicate the color of the sample. Sub-numerals on Fig. 3 ($12Ab_1$ and $12Ab_2$) represent two materials from the same manufacturer and having the same nominal thickness and color. The sequence of bars representing results obtained with different electrodes and media is indicated for group $5Bb$ of Fig. 3 and is consistent for subsequent groups of both charts. The bars represent average dielectric strength of dry specimens and the shaded portions represent dielectric strength of moisture-conditioned samples. Since twenty-five breakdowns were taken in each instance for a single arithmetic average, it is seen that the charts, Figs. 2 and 3, are a compilation of 6400 individual dielectric breakdowns.

An examination of these charts showed certain effects to be consistent for the various samples. It is evident that bars representing results with dry samples followed a definite pattern with electrode and media used. The pattern persists for the thicker moisture-conditioned samples but is somewhat disturbed for the thinner materials.

The general trend toward lower dielectric strength with increased thickness is evident for dry samples, if 5-mil bias-cut samples are excluded, while a reverse effect is also evident for moisture-conditioned samples. It is also noted that the yellow samples disturbed these trends by yielding generally lower results, but this effect cannot be used as a criterion, since yellows had been supplied by only one manufacturer.

Since it was not possible to obtain samples of the four desired thicknesses and of both colors and cuts from all manufacturers, a considerable unbalance of data resulted. Due to this unbalance, an indiscriminate comparison of all results would lead to erroneous conclusions. It would be misleading, for example, to compare averages of all bias-cut samples with all straight-cut samples when thicknesses, manufacturers, and colors were not equally represented in the two groups; or to determine effect of thickness using all samples when representation of the four nominal thicknesses varied to so great a degree. Furthermore, an attempt was made to evaluate the interactions among factors to enable a quantitative determination of effects attributable to specific factors. Therefore, in making each analysis, the results were arranged in balanced groups and these were then studied, using recognized statistical procedures, to determine the effect of a particular factor. Such analyses were used in drawing the conclusions which follow.

CONCLUSIONS

Dielectric strength after moisture conditioning in general was about 75 per cent lower than the dry dielectric strength. This difference varied with thickness, moisture showing less effect (about 60 per

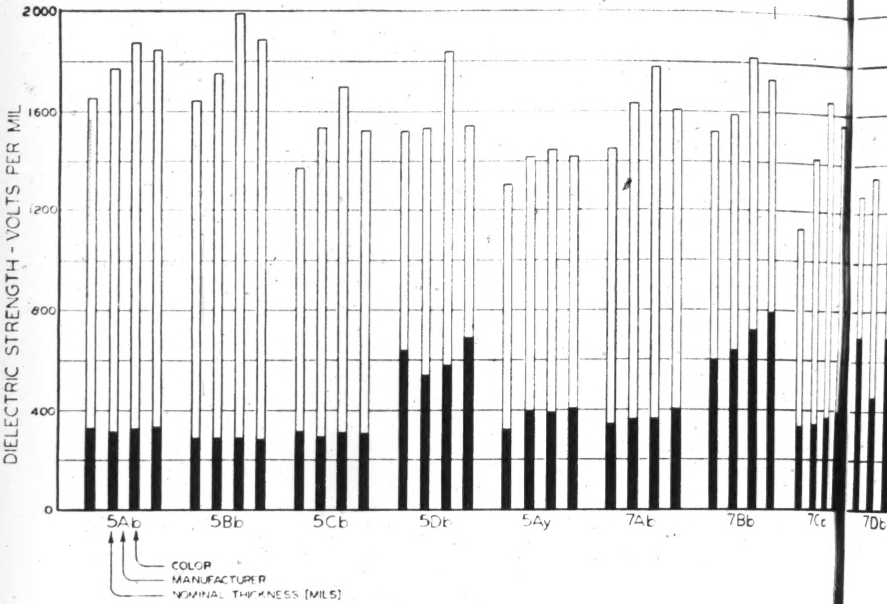


FIG. 2.—Dielectric Strengths Straigh

The average dielectric strengths of dried specimens are represented total ba
shaded sections. Twenty-five breakdowns were used to obtain each avera

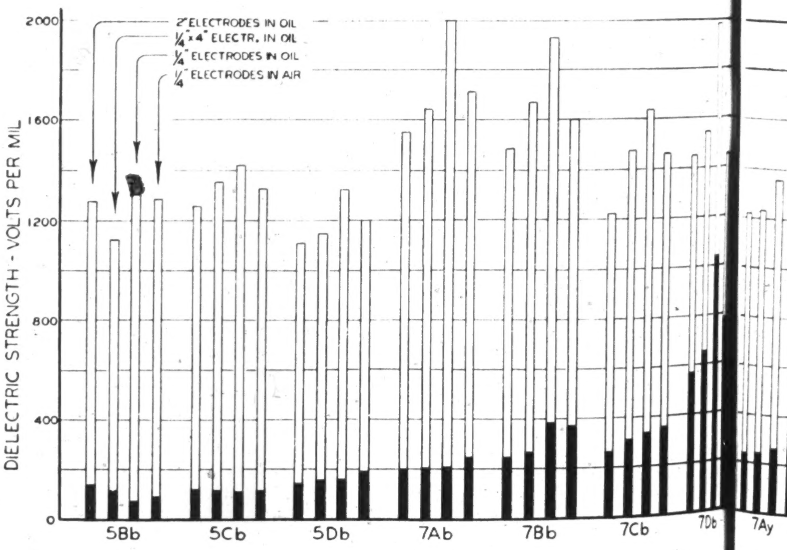
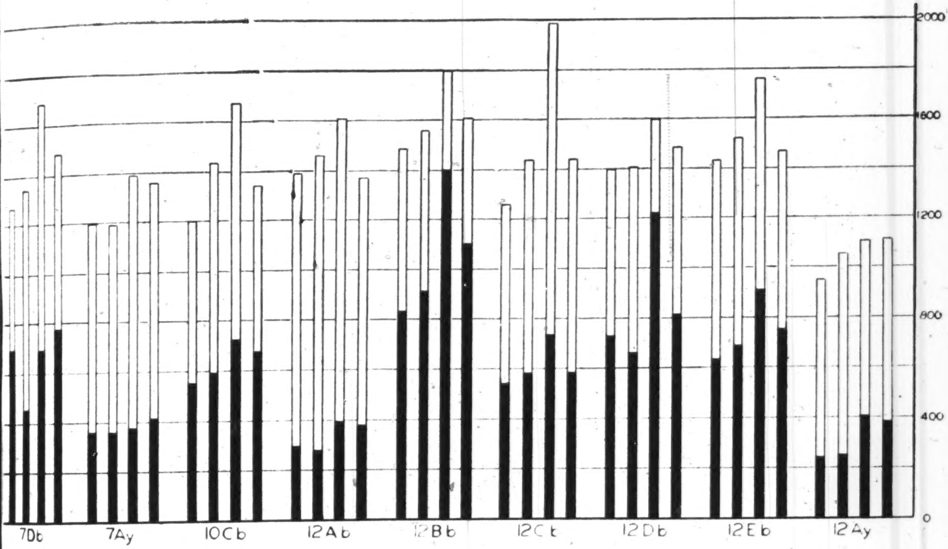


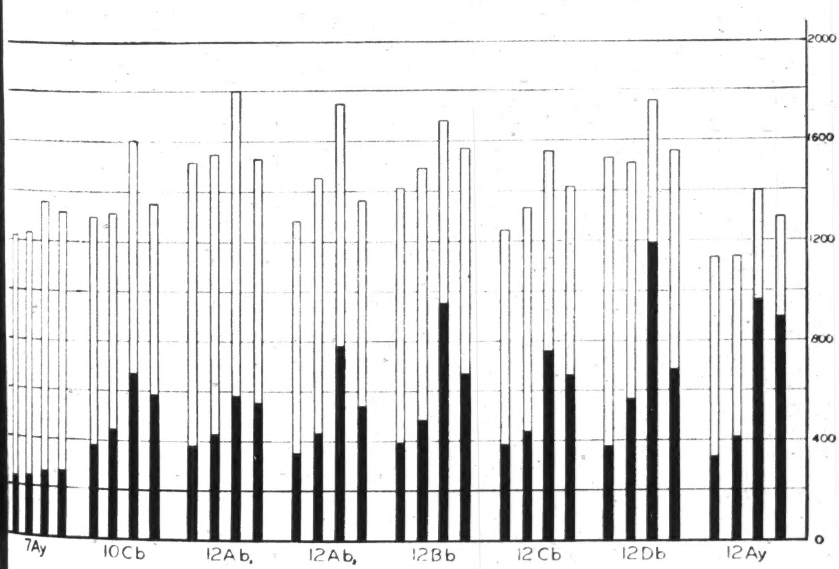
FIG. 3.—Dielectric Strengt of Bias-

The average dielectric strengths of dried specimens are represented total bar
shaded sections. Twenty-five breakdowns were used to obtain each avera



Straight-Cut Varnished Cambrics.

total bar heights and those of high humidity conditioned specimens, by heights of



of Bias-Cut Varnished Cambrics.

total bar heights and those of a high humidity conditioned specimens, by heights of cent) on the thicker materials. When considering 12-mil black materials only, it was noted that the bias cut showed a greater effect of exposure to high humidity than the straight cut. There was considerable difference in the effect of humidity on samples from the various manufacturers. On 12-mil material, manufacturer *B* showed the least loss (about 34 per cent) while manufacturer *A* showed the greatest (about 78 per cent), indicating that variations among cuts and thicknesses of cloth would be considerably less than might be expected among manufacturers.

In comparing effect of electrodes and using the 2-in. electrodes as a reference base, the $\frac{1}{4}$ by 4-in. gave approximately 5 per cent higher dielectric strengths and the $\frac{1}{4}$-in. showed results about 27 per cent higher. These variations were more accentuated on the bias than on the straight-cut materials. Increase of measured dielectric strength is of course to be expected with decrease of electrode size. However, it appears from the above that changes in electrode size, as might be caused by ionization of surrounding air, will produce considerable effect on measured dielectric strength with the $\frac{1}{4}$-in. diameter electrode, but little effect with the $\frac{1}{4}$ by 4 in. In other words, a curve of dielectric breakdown *versus* electrode diameter would be very steep at $\frac{1}{4}$ in., but would have flattened out considerably at $1\frac{1}{8}$ in. (approximate circular equivalent of $\frac{1}{4}$ by 4-in. electrode). It is believed, therefore, that results obtained with the larger electrodes would have more value.

Dielectric breakdown measured in air appeared to be about 9 per cent lower than when measured in oil. This conclusion is based only on results obtained with $\frac{1}{4}$-in. diameter electrodes. As might be expected, the difference varied with thickness of specimens, increasing as thickness increased.

It should be noted that the scope of the investigation was sufficiently broad to permit studies of factors other than those discussed earlier. An analysis showed, for example, that in general the average dielectric breakdown of bias-cut cloths was about 25 per cent lower than for straight-cut materials. However, a further analysis of presented data was considered too digressive for this article.

It was also recognized that the investigation itself might have followed more extensive lines. For example, it might have been possible to include round electrodes of several diameters but having the same contact edge curvature, so that the effect of electrode area could be studied. It is apparent that such a study would have involved dry and high humidity conditioned samples of several thicknesses, in air and in oil. Likewise, with electrode area held constant, an investigation of the effect of contact edge curvature could have been included. To restrict the scope of the work to some practical limits, methods believed to be commonly used were studied, as was mentioned previously.

The authors favor the use of 2-in. diameter electrodes because they provide adequate sample coverage with reasonably large contact edge curvature, minimize alignment errors, and may be readily resurfaced. Furthermore, during breakdowns in air, ionization may cause an increase in what might be termed "effective electrode area" and, since this change is dependent on voltage, the net effect is analogous to larger electrodes at high breakdowns than at low breakdowns. It is evident that the smaller the electrode used, the greater the per cent change of "effective electrode area" with voltage. Where use of 2-in. electrodes is not possible, as in testing tapes, this investigation has indicated that the $\frac{1}{4}$ by 4-in. electrodes may be substituted with little difference in results. However, substitution of oil for air medium, or *vice versa*, does not appear feasible, since results are comparable only at low breakdown voltages, those in air tending to be much lower as breakdown voltages increase. The two humidity conditions were readily maintained and are considered suitably indicative of the effect of high humidity on varnished cambric. In this respect, the use of small chambers, such as glass desiccators, is preferred to large cabinets for maintaining condition and for handling groups of specimens.

In concluding, the authors express the hope that this investigation has attained its objective in presenting a quantitative study of certain factors which commonly affect the determination of dielectric strength of varnished cambric cloth.

Acknowledgment:

The authors wish to acknowledge with appreciation the service of M. Zangrillo for assistance in the collection of data, and of D. R. Embody and his staff for statistical analyses.

THE THEORETICAL BASIS OF ADHESION[1]

By W. A. Weyl[2]

Problems of Adhesion in Glass Technology

My being introduced to you as a glass technologist raises the question: What has glass technology in common with adhesives? There are quite a few cases where a research worker in the field of glass and one in the field of adhesives have a common interest in the fundamental question: What causes two materials to stick together? Before giving you more concrete examples for adhesion problems in the field of glass technology, let me tell you how I became interested in the basic phenomena of adhesion.

Several years ago I studied the influence which halogen ions, like bromine and iodine, exert on the color of metal-ion-containing glasses. For this purpose low-melting borates of alkali were mixed with various metal oxides; for instance, copper oxide and bismuth oxide, and the mixture was fused in a platinum crucible. The melt was then poured into a mold for further examination. As usual in these cases, the platinum crucible was covered with a layer of glass and had to be cleaned before the next experiment could be made. On repeating these experiments with the addition of a few per cent of potassium iodide to the glass batch, I was surprised to find that now the melt could be poured out to the last drop. The fused borates behaved like mercury in a glass container; they did not show the slightest sign of wetting. If a part of the melt was left in the platinum crucible on purpose, it solidified to a glass which did not show adhesion to the metal, but came out in one piece. Such behavior was very unusual and invited further study (**1**).[3] In the course of these investigations, it was found that replacing the oxygen ions of a glass by other anions had a decisive influence on its adhesion properties. In some cases it decreased adhesion to the platinum, as mentioned before; in other cases where sulfur was substituted for oxygen of a phosphate glass, a strong increase of adhesion to steel was observed. The "affinity" of the anionic constituent of a glass to the metal seems to be a major factor in determining the adhesion. I use the word "affinity" to describe the cause of adhesion because I assume that it is fairly well established that the adhesion of two materials, even between glue and porous wood, is primarily a chemical phenomenon. In some cases, no doubt, the gripping quality of the surface might favor the adhesion, but as a rule the mechanical contribution seems to be negligible.

If we accept a chemical picture of adhesion, then we realize at once that understanding the problems of adhesion requires the knowledge of the nature of chemical forces in general, and in addition to that, information about the surface of the materials to be combined.

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[1] Presented at a meeting of Committee D-14 on Adhesives, held in Buffalo, N. Y., June 27, 1946.

[2] The Pennsylvania State College, State College, Pa.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1516.

It is the purpose of this paper to discuss these two factors and to select examples from my special field. I do that, not only because glass technology is the field with which I am most familiar, but also because recently R. C. Rinker and G. M. Kline, (2), of the National Bureau of Standards made a very valuable contribution by their more general "Survey of Adhesives and Adhesion" for the National Advisory Committee for Aeronautics.

The field of glass technology offers a great number of examples in which adhesion of glasses to other material becomes an important factor. Perhaps best known is the combination of glass with organic high polymers, as used in the manufacture of safety glass and for a large number of fiber-glass products. There are also numerous examples, especially in the electric industry, in which glasses and metals have to be combined at high temperature. The low-temperature adhesion of metals to glass is required in the manufacture of mirrors and for other types of decoration. During the war another field has been developed where glasses are coated by inorganic compounds, such as fluorides, oxides, and sulfides, in order to change the reflectivity of optical elements. All these different techniques have in common the problem of adhesion. Research workers in these branches are confronted with questions such as the following: Which organic high polymer has the best sticking quality to plate glass? Our Decoration Department has difficulties with the adhesion of gold on tableware, what can be done about it?

The whole enamel industry is based on the adhesion of certain low-melting glasses to metals. The glass industry is also interested in the reverse side of the problem. The sticking of glass to the metal of molds has to be avoided. The same forces which are responsible for jewelry enamel sticking to gold or copper are also responsible for the solubility of these metals in glass melts. That brings a whole group of colored glasses, namely, copper ruby, silver stain and gold ruby, close to our problem.

This short excursion into the field of glass technology will indicate why a glass technologist can become interested in the problem of adhesion.

THE NATURE OF CHEMICAL FORCES

Surface Forces:

The modern concept of the chemical forces acting among different atoms or molecules is based on the distribution of their electrical charges. In the first approximation, one can treat most chemical forces on the basis of electrostatic attraction and repulsion. The intensity of the force is governed by the elementary laws of electrostatics where charge and distance are the dominating factors. We must bear in mind, however, that picturing an ion as a rigid sphere represents an over-simplification. Large ions, especially those with incomplete outer electronic shells, may easily change their shape and dimensions under the influence of the electric field of neighboring ions, so that they can no longer be treated as spheres.

A substance like a soda-lime silicate glass, or a simpler compound, such as sodium chloride, consists of electrically charged cations and anions which are arranged in a way that each cation is surrounded by a certain number of anions, and each anion is surrounded by cations. The attraction between ions of opposite charge and the repulsion of ions having the same charge lead to a configuration which might either consist of self-repeating units (crystal structure) or of a random three-dimensional network (glass). As far as the nature of the forces is concerned, there is no fundamental difference between crystals, glasses, and liquids.

It is important to realize that a crystal of sodium chloride does not consist of an accumulation of NaCl molecules, but, instead, of a three-dimensional network where each sodium ion is surrounded by precisely six chlorine ions, and each chlorine ion is surrounded by six sodium ions. The modern picture of the structure of solids has no use for the old-fashioned concept that the number of valences of an element can be pictured by a corresponding number of hooks. Sodium, for instance, has only one valence, but in the structure of sodium chloride it divides its attraction force equally among its six neighboring chlorine ions. This difference between the old concept of valence and the modern approach is important for us because it will explain the forces emanating from a glass or crystal surface. According to the old concept, the "chemical affinity" of a given number of chlorine atoms is completely satisfied if we add to it precisely the same number of sodium atoms. A compound of the formula NaCl is considered saturated because the two monovalent elements have combined in the ratio 1:1.

The modern concept, however, leads to the conclusion that this does not hold true for the surface. Saturation of the electrical forces exists in the interior of the crystal, but in the surface layer some ions obviously cannot be completely surrounded by their partners. This leads to a state of unsaturation. In order to keep the number of unsaturated ions at a minimum, the substance exhibits a tendency to decrease its surface area (surface tension). Mercury, for instance, tries to assume a spherical shape because a sphere has the smallest surface area for a given volume. Finely subdivided crystals have a tendency to recrystallize, that is, to decrease their surface area by forming a smaller number of larger crystals. A liquid mixture has still another possibility of decreasing its surface energy. Those molecules which decrease the surface tension accumulate in the surface film. Capillary active substances, such as higher alcohols, fatty acids and organic amines, decrease the surface tension of water and, as a result, migrate into the surface layer. The same process takes place in glasses at elevated temperature. It causes the alkali ions with their one-fold positive charge to migrate into the surface, whereas the strong Si^{4+} ions try to remain in positions where they still can be completely surrounded by the anions O^{2-}.

Boric oxide-containing glasses exhibit a strange phenomenon. Upon the addition of increasing amounts of boric oxide to an alkali silicate glass, all properties go through extreme values. Density and refractive index, for instance, show maxima. Thermal expansion and electric conductivity show minima. This influence of boric oxide has been called the "boric acid anomaly" by glass technologists. It is due to the fact that the B^{3+} can form two types of anions: $(BO_4)^{5-}$ and $(BO_3)^{3-}$. This change of coordination, or the transition from BO_4 tetrahedra into BO_3 triangles, accounts for the change in the properties. It is significant that all properties show this anomaly with the exception of the surface tension. The surface of these glasses never contains BO_4 groups, but only BO_3 triangles.

There still exists another possibility for many solids to decrease the surface-free energy which is often neglected, but is very important for adhesion problems. A substance may saturate its residual forces by adsorbing other molecules from the atmosphere. We rarely deal with "clean" surfaces.

Most surfaces are contaminated. The atmosphere contains both water and minor amounts of organic matter. We all know how difficult it is to clean a glass plate so that it is wet by water evenly and completely. But even if the surface of a glass is free from grease, it will still be different from the bulk of the glass. It contains OH groups from adsorbed water molecules which have reacted with the glass and caused hydrolysis.

Liquids, crystals and glasses have surfaces which may differ considerably from that which we would expect from the known structure of the bulk of these substances.

Some Types of Adhesion to Glass:

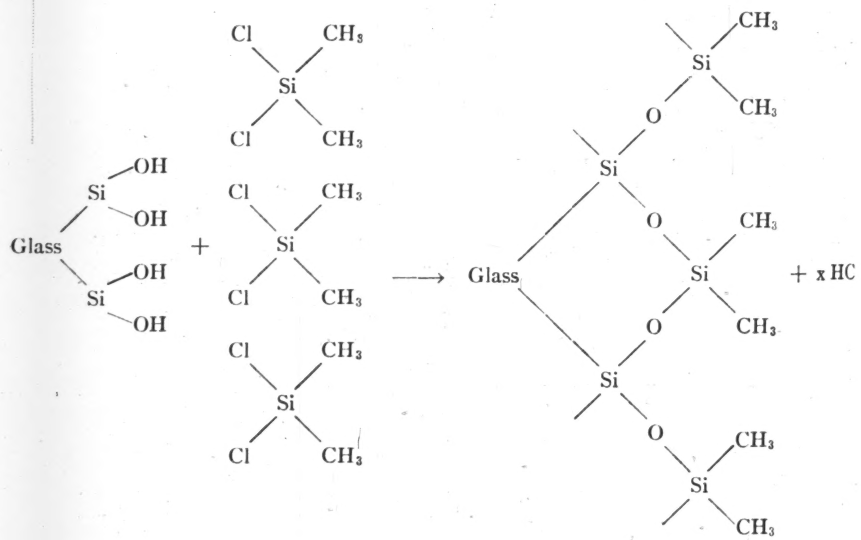
A few examples of adhesion to a glass surface may illustrate the different types of chemical forces responsible for this phenomenon.

Continuation of the Glass Structure with Other Building Units.—We can describe the forces acting on the surface of a glass or of a crystal as its endeavor to continue its structure. The recrystallization of finely precipitated barium sulfate in contact with its saturated solution, for instance, is due to the tendency of some crystals to grow at the expense of others. The same phenomenon occurs not only through a liquid, but also through the vapor phase if the substance has a noticeable vapor pressure. It can occur in solid phases if the diffusion speed of the units is sufficiently high. Recrystallization is caused by the tendency of a surface to continue the crystal structure by means of adding the same building units. If the same building units are not available, the structure might be continued by other compatible units. The seeding of super-cooled solutions by an isomorphous substance and the oriented growth of potassium iodide on mica are typical examples. The influence of surface forces on the crystallization from saturated solutions can be so strong that it can produce a crystal lattice which is thermodynamically unstable, only because its parameters fit better to the seeding surface than those of the crystal which one would expect from the equilibrium diagram.

Continuation of the glass structure with other building units is responsible for the adhesion of crystals such as magnesium fluoride or calcium fluoride when these substances are brought onto the glass surface from the vapor phase. The structures of these crystals are compatible with that of the glass. They are built from similar ions and the salts are soluble in the glass melt. The principle of continuation of a structure with other compatible building units is responsible for many adhesion phenomena.

Chemical Reactions of the Glass Surface.—Building up the structure of a glass surface with compatible but different units can take place only under conditions (vacuum and elevated temperature) where the surface of the glass is clean. A glass surface does not remain like that very long when it is exposed to the atmosphere. It attracts water molecules and hydrolyzes. The surface, therefore, does not contain oxygen ions, but extends OH groups into space. These hydroxyl groups are responsible for the adhesion of glass to many organic compounds, such as dyestuffs, high polymers and animal glue. One must not think of these forces as secondary forces which are weaker than those acting within the materials. Animal glue with its reactive carboxyl and amino groups can interlink with the hydroxyl groups and the alkali of the glass in a way that the shrinkage of the glue under the influence of heat tears out pieces of glass rather than break at the glue to glass interface. This phenomenon has been used commercially for many years to produce a type of frosted glass. This method of decorating glass is based on the fact that the forces acting between glass and glue are stronger than the cohesive forces acting within the glass.

In order to learn more about the nature of adhesion, another modern technique is very instructive: namely, the reaction of a glass surface with organic silicones. Compounds of the type of dimethyl silicon chloride $Cl_2Si(CH_3)_2$ are used commercially to make a glass surface water repellent by imparting to it the hydrophobic properties of hydrocarbons. This is accomplished by exposing the glass to the vapors of organic silicon chlorides at elevated temperature. The reaction can be describedd by the following:



The strong adhesion of "Dry Film" to glass or ceramic materials is due to a chemical reaction. Many cases of adhesion can be described by similar chemical reactions.

The structure of a glass surface is closely related to that of an important adhesive: water glass. Water glass is a highly polymerized silicate of sodium or potassium which is used in aqueous solutions. Its adhesion properties are caused by the reactivity of the Si-O-Na groups. This brings up an interesting question: Why do we use water glass as an adhesive and not a solution of silicic acid or silica gel? Colloidal silica does not have the sticking quality of a water-glass solution. It ages because its free reactive hydroxyl groups react with themselves, thus increasing the molecular weight of the substance and decreasing its chemical reactivity. One of the requirements of a good adhesive is the presence of reactive groups under conditions which prevent these groups from reacting with themselves. The hydroxyl groups of a silica gel have a tendency to split off H_2O and to form a permanent bond. This is less likely to happen if we use water glass which does not have free Si-OH, but Si-O-Na groups. Nevertheless, these groups hydrolyze readily in contact with other materials, such as cellulose, and enter similar reactions as a glass surface.

The phenomenon that active groups may react with themselves is perhaps the most serious limitation in the "construction" of new adhesives based on chemical principles. For example, if we want to increase the adhesion between a glass and a high polymer, the logical step would be the introduction of stronger polar and more active groups into the high polymer. Unfortunately, the presence of too many polar groups in a plastic would not only increase the forces between the organic medium and the glass, but also would increase the forces between the organic molecules themselves. The latter makes the high polymer stronger perhaps, but decreases the plasticity in such a way that it becomes brittle.

The interaction and adhesion of molecules with OH groups is noticeable even in the liquid state. Water itself can be considered strongly associated. Recently water has been supercooled to a temperature of −72 C. where it forms $(H_2O)_6$ (3). In order to understand completely the properties and the forces among these polymers, one does well to use a dynamic picture rather than the static one which we derived from chemical bonds. The high viscosity of sugar syrup and glycerol is the result of a high degree of association due to the attraction between OH groups.

Adhesion Caused by van der Waal's Forces.—Up to this point we have treated adhesion as a formation of new chemical bonds. The continuation of the glass structure by other building units or the coating of glass with glue and "Dry Film" are typical chemical reactions which can be described by means of chemical equations. There are, however, a number of cases where truly chemical reactions must be excluded as a possible explanation for the adhesion. The forces between neutral atoms or molecules are often summarized under the name of van der Waal's forces. We will now investigate the forces which result from a neutral atom or molecule approaching the surface of a crystal or glass.

(*a*) *Attraction of a permanent dipole.*—Many molecules, even when neutral in themselves, exert electrical forces because their electric charges are not symmetrically distributed. Such a molecule is called "polar" and its polarity is measured by its dipole moment. At high temperature, when the molecules of vapors or liquids rotate, one cannot expect much interaction between them and the electric forces emanating from the surface ions of crystals and glasses. As long as the molecules rotate, positions leading to attraction and repulsion become equally probable. If thermal motion decreases, rotation ceases and changes into oscillation about equilibrium positions. Now the molecules orient themselves with their positive parts toward the negatively-charged ions of the surface and attraction results. Adhesion of a molecule due to orientation in the electric fields of the surface increases with decreasing temperature, increasing polarity and size of the molecule. Orientation and attraction of polar molecules are often the first steps to adhesion. The oriented molecules may later react chemically with the surface.

(*b*) *Attraction of an induced dipole.*—P. Debye pointed out that not only rigid dipoles but also nonpolar molecules can be oriented and attracted by an electrically-charged particle, if they are sufficiently polarizable so that a dipole moment can be induced. Regardless of the way the molecules are oriented, polarization always leads to attraction and never to repulsion. As a result, these forces are less subjected to the influence of temperature and thermal vibration. They, too, increase with increasing polarizability and increasing size of the molecule.

(*c*) *Attraction caused by dispersion forces.*—According to classical physics, there should be no attraction between two neutral atoms. Nevertheless, spectroscopy reveals that short-lived compounds exist between atoms, such as cadmium and helium. We have little exact knowledge about the nature of these dispersion forces, but again the polarizability plays an important rôle. The dispersion forces are fundamentally different from those acting between permanent or induced dipoles. They are strictly additive and depend only on the distance of the molecules and not on their mutual orientation or on the surroundings.

Polarization—a Major Factor in Adhesion.—In our concept regarding the attraction forces between neutral atoms or molecules, we have used polarizability and polarization as a major factor. For many years K. Fajans (4) and his students called attention to the fact that it is not permissible to interpret the interaction between two ions on the basis that each ion has a definite size. The distance between silver ions or sodium ions respectively, and different halogen ions, offers a striking example. Comparing the cation-anion distance which is available very accurately from X-ray diffraction of the sodium and silver halides, we find that sodium and silver ions have about the same size in the chlorides. Assuming that the halogen ions would have a constant size, we must come to the conclusion that the silver ion is larger than the sodium ion in the fluorides. In the bromides, and even more so in the iodides, the silver ion seems to be much smaller than the sodium ion (Table I). These and many similar facts prove that ions cannot be treated as rigid spheres. Especially large ions are easily deformed by their neighbors. The electron clouds surrounding the positive nuclei become asymmetrical and the attraction forces are increased. Small ions with high charges, especially those with incomplete outer electronic shells, exert stronger deforming or polarizing influences than large monovalent ions of noble gas structure. It is not possible here to discuss in detail the Fajans' theory of the polarization of ions, but let me call your attention to his Baker Lecture (4), where the chemical forces are discussed on this basis. A few typical examples will serve to illustrate the usefulness of this concept for better understanding adhesion forces.

TABLE I.

Cation	Interionic Distances for Various Halides, 10^{-8} cm.			
	F	Cl	Br	I
Ag^+	2.58	2.78	2.89	2.83
Na^+	2.32	2.81	2.98	3.23
Difference	+0.26	+0.03	−0.09	−0.40

EXAMPLE I: STABILITY OF MOLECULAR FILMS OF ALKALI METALS

For neutral atoms of comparable electronic configurations the polarizability and, therefore, the adhesion increases with increasing size. This is brought out in the work of A. C. B. Lovell (5), who studied the stability of molecular films of alkali metals on glass surfaces *in vacuo.* The stability of these invisible films was determined by the change of their electric conductivity with time. Films which consist only of a few atomic layers have a tendency to deteriorate rapidly through recrystallization and thus to increase their electric resistance. Cesium films were found to be the most stable. The stability of the alkali metal films decreased in the following order: cesium, rubidium, potassium. This indicates that the adhesion of neutral alkali atoms is a function of their polarizability which increases with the size.

EXAMPLE II: GILDING AND SILVERING OF A GLASS SURFACE

Glass tableware made from a soda-lime glass is not very suitable for gilding. The absence of polarizable ions is responsible for the failure of the gold film to stick to the glass surface. Experience has taught the decorator that good sticking can be achieved by "mudding" the glass, that is by treating the parts to be gilded with a mud or slip made of clay, ocher and silver or copper compounds. This operation carried out below the softening point of the glass modifies its surface and introduces silver or copper ions which have nonnoble gas structures. Their presence promotes adhesion to the noble metal. The same is true for the silvering process, where either Sn^{2+} or Ti^{3+} ions can be used to promote adhesion.

Adhesion of a silver mirror to soda-lime glasses is promoted by a previous treatment of the glass surface with tin compounds. Tin ions help to bridge over the incompatibility of silver and glass. Their effect is not restricted to soda-lime glasses, but they accomplish the same adhesion between silver and plastics.

EXAMPLE III: ADHESION-PROMOTING IONS IN ENAMELS

Enamels are low-melting glasses of the borosilicate type which are used as coatings for metals such as noble metals (jewelry), steel (kitchen utensils) or cast iron (bath tubs). Adhesion to the metal is due to one of the following reasons:

(*a*) The presence of lead ions as a major constituent in the enamel.

(*b*) The presence of a specific adhesion-promoting ion of the transition group: Ni^{++}, Co^{++}, Fe^{++}, Mn^{++}.

(*c*) The presence of a large polarizable ion: Sulfur. (Phosphate glasses have been made adherent to metal by the addition of sulfides.)

EXAMPLE IV: SOLUBILITY OF NOBLE METALS IN GLASS

For the manufacture of certain colored glasses such as silver yellow and gold or copper ruby, it is essential that the base glass exerts some solvent power upon the metal. As long as heavy lead glasses were used, no difficulties were encountered. Soda-lime glasses can be used, however, only if small quantities of tin oxide or bismuth oxide are introduced. Metallic gold is soluble only if ions of sufficient polarizability are present. Recently a glass melt of good solvent power for platinum was produced from noble gas ions only. This glass contained the large polarizable cesium ion as a major constituent. In this case the size of the cesium ion permitted the interpenetration of electrons essential for the solubility of metals.

Solvation forces and adhesion forces are similar in nature, so that insight into adhesion problems may be gained from solubility studies. The same ions which promote adhesion between a glass surface and a metal were found to promote solubility of metals in glass.

These few examples will serve to demonstrate the usefulness of Fajans' theory on the mutual polarization of ions for interpreting the nature of adhesive forces. The strange phenomenon which we described at the beginning of this paper, namely, the lack of adhesion to platinum of certain borate melts with minor additions of iodide can be explained on the same basis. The large iodide ions are strongly deformed by cations such as Pb^{++}, Bi^{+++}, and Tl^{+} much more so than the oxygen ions whose places they occupy. This deformation leads to strong asymetrical forces between the anion and the cation. The residual forces emanating from the anion are consequently only weak. As a result, the molecules of PbI_2, TlI or BiI_3 are pushed to the glass metal interface where they weaken the adhesive forces between the glass melt and the platinum metal of the container wall. Failure to wet the wall and lack of adhesion are the results.

We might mention here that quite frequently the glass technologist has to be interested in the problem of avoiding adhesion. All commercial glasses have oxygen ions as the anions. Oxygen has a strong affinity for all metals. Even platinum is wet by glass through the strongly adhering layer of oxygen. B. S. Ellefson and N. W. Taylor (6) demonstrated that *in vacuo* or in nitrogen where the oxygen layer on the platinum is absent, a silicate glass does not wet the metal.

If the glass technologist wants to prevent sticking of the hot glass to the metal of molds or machine parts, he lubricates the molds with graphite or carbonaceous materials. Under these conditions the oxygen of the glass cannot adhere to the mold by means of oxide formation. The reaction product, carbon monoxide, is a gas.

METHODS OF STUDYING SURFACE FORCES

We have learned that a surface emanates specific forces which can be greatly modified by adsorption of water and organic matter from the atmosphere.

In some cases the change of the surface through adsorbed molecules favors adhesion; in others, it brings about the reverse effect. This raises the question of how to learn more about the surface forces and how to analyze and estimate them. It is obvious that there is no best method to accomplish this aim but, depending on the nature of the substances which we want to join, different specific methods have to be chosen.

Wetting and Contact Angle:

For more than one hundred years one has known that the shape of a drop of a liquid placed on the smooth surface of a solid is related to the forces acting between the three adjacent phases: solid, liquid, and gas. Contact angle measurements have been widely used to learn more about these forces and many experiments met with considerable practical success.

We mentioned previously the work of Ellefson and Taylor (6) who measured by means of the sessile drop method the adhesion between glasses and metals as a function of the temperature and atmosphere. C. M. Lampman (7) used the contact angle to judge the wetting ability of various ceramic bodies by glazes.

In our laboratory H. H. Kellogg developed a method for studying the nature of glass surfaces which were in contact with aqueous solutions of different salts. We know from experience that certain ions, such as Al^{3+}, are very strongly adsorbed at a glass surface and that the chemical properties of such a surface will then be modified.

For studying these adsorption phenomena, lauryl trimethyl amine bromide was used as an indicator. Traces of this organic compound when adsorbed at the surface will make the glass water-repellent. Competition between the organic amine and other ions present in the same solution permits us to judge the affinity of various ions to the glass surface. The degree of adsorption of the lauryl trimethyl ammonium ion is measured by the degree of water repellency. The latter was measured by observing the contact angle of an air bubble pressed against the glass which was immersed in a solution containing both the indicator and the ion under examination. This method has been developed by Taggart (8) for studying the mechanism of mineral flotation. The contact angle method can be modified in several directions and will no doubt remain a very useful tool for studying surface forces.

In an entirely different form we can use the contact angle to judge the affinity of a surface to a liquid. We are familiar with the condensation of water vapor upon surfaces of glass or metals. The uniformity of the dew deposited from the breath is commonly used for judging the cleanliness of a glass surface. The breath figures are indicative of the competition between the molecular film of grease on a glass surface and the water which tends to wet completely the surface. The observations and discussions on this subject made by Lord Rayleigh (9) may stimulate further thoughts and research in this direction. The optical effects of these little droplets can be measured by their characteristic scattering. The solid need not necessarily be glass, but metals could be used, and the scattering of the reflected light be measured. Instead of water vapor, the vapor of various organic compounds could be condensed and in this way the "breath figure" method could be adapted to various needs.

Friction Between Solids:

Another possible method for studying the replacement of one molecular species by another is given by the use of "static friction" as an indicator. The origin of some of the best work on adhesion and on spreading of fluids on glass surfaces can be traced to some thoughts which Lord Rayleigh had on the slipping of a cup of tea about its saucer. Probably everybody has noticed that slipping can be prevented if the parts in contact are wetted with a drop of tea. This paradoxical behavior of the porcelain, that lubricating the solid phases by wetting with water increased friction, led to a thorough examination of friction between glass surfaces. W. B. Hardy and J. K. Hardy (10) measured the force required to move a loaded watch glass over a polished glass surface. No matter whether they used optically polished glass or plate glass, the force was relatively great if the surfaces were thoroughly cleaned. The clean surfaces could not slide over one another. Under the slightest pressure they seize and grow together. Pulling the watch glass over the surface produces a sequence of fractures as can be seen under a high power microscope. The track of the loaded watch glass is about 50μ wide and consists of fractured glass. A molecular film of oil prevents seizing and decreases friction. Water is not as effective, but it adheres more strongly to a clean glass surface. Based on these experiments, a simple explanation could be given for the teacup experiment. The sliding is caused by a film of grease which adheres to the surface of cup and saucer. Wetting with water replaces the lubricant and friction increases.

Surface Diffusion of Gold:

Sometimes it becomes necessary to work out very specific methods for judging the adhesion forces existing between two materials. As an example, I will mention a method which we developed in our laboratory for the specific purpose of learning more about the forces acting between noble metals and glasses or ionic crystals. This method is based upon the aggregation of noble metal atoms which can be easily followed by the change in light absorption and fluorescence. The first step consists of the adsorption of gold or silver ions on the surface to be studied. The second step, either thermal dissociation or reduction by hydrogen gas, changes the ions into neutral atoms. The atomic subdivision of both metals is colorless, but in the case of silver may produce a white fluorescence on exposure to ultraviolet radiation. The atomic subdivision of a metal is not stable. The atoms try to aggregate and to form stable crystals. This is accomplished by surface diffusion and collisions. Surface diffusion is delayed by strong forces acting between the atoms and the surface of the carrier. The stronger the forces, the higher will be the temperature required to bring about aggregation of the metal atoms. The final step in this process consists of heating the substance covered with metal atoms for a certain length of time to various temperatures. The progress of the aggregation can be followed by means of the colors produced. Gold crystals of colloidal size appear pink. On further growth they change to purple and, finally, if the crystals are large in size but small in number, the color begins to fade. Silver atoms lose their fluorescence and develop yellow to brown silver crystals.

Experiments with this surface-diffusion method brought out the fact that noble gas ions exert only weak forces upon the gold atoms. Nonnoble gas ions, due to their greater polarizability, prevent aggregation, indicating that the adhesion forces become of the same order of magnitude as the cohesive forces in the gold crystal.

SUMMARY AND CONCLUSIONS

Adhesion is a complex phenomenon

and should not be attributed to one particular cause. In most cases the contribution of a purely mechanical gripping quality of the surfaces is negligible, and adhesion results from chemical forces. For many adhesion processes the mechanism of bond formation could be described by means of chemical equations.

The van der Waal's forces which are small for most molecules can reach the strength of chemical bonds if we deal with large, and especially with asymmetrical, molecules.

Understanding adhesion phenomena requires knowledge of the nature of the surfaces which have to be joined and of the nature of the chemical or electrical forces acting between the two phases.

As far as the surface is concerned, one has to realize that its structure can be very different from that of the bulk of the substance. The reasons for structural changes in the surface are the migration of capillary active molecules into the surface layer and adsorption of water and organic matter from the atmosphere.

As far as the forces are concerned, one has to consider the difference between an ion in the interior of a crystal or liquid, where it is more or less symmetrically surrounded by its partners, and the asymetrical forces acting in the adhering layer. Under the influence of a one-sided attraction and repulsion, the ions will be more strongly deformed or polarized than in the crystal. The polarization forces become a major factor in adhesion. Several examples are given which show the importance of the polarization of ions and atoms for the solubility of metals in glass and their adhesion to glasses and crystals. The outstanding role played by the large and easily deformable anions, such as sulfide, and of the strongly polarizing cations of nonnoble gas character, finds its explanation on the basis of Fajans' theory.

A few general and one specific method for studying the forces acting between surfaces and vapors, liquids and solids are discussed. There are methods which can be adapted to specific problems in research on adhesives.

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DISCUSSION

Mr. A. J. Warner.[1]—It is well known that polyethylene wets metals with great difficulty and it has been particularly difficult to get any adhesion between polyethylene and the copper wire normally used as the conductor in high-frequency cables. It has been observed, however, that if the copper is oxidized so that a substantial oxide film is present, then polyethylene will adhere and give a relatively strong bond. Perhaps Mr. Weyl may care to comment on this.

Mr. Weyl (*author*).—The first step in the oxidation of metallic copper leads to the cuprous oxide Cu_2O. This compound has lattice parameters which do not differ much from those of the metal. As a result, the structure of the metal is continued in the oxide layer, and one can expect strong adhesion between both phases. The fact that polyethylene adheres strongly at the oxide might be attributed to the formation of a chemical compound. Cuprous salts are known to form complexes with ethylene. W. Manchot, for example, isolated a compound between cuprous chloride and ethylene. From this point of view it would be interesting to examine the adhesion between polyethylene and metallic copper which had been exposed to halogen vapor instead of oxygen.

Mr. R. E. Parkinson.[2]—Would Mr. Weyl explain the sticking of metal to glass at high temperatures, such as occurs in an overheated glass mold? This sticking takes place at approximately the same temperature for different alloys and is different from the phenomenon of ad hesion where high temperatures cause a weakening of the adhesive bond. Is there any way to suppress this tendency to stick?

Mr. Weyl.—All metals, even platinum, are covered with an oxide layer. At a high temperature this oxide reacts with the glass, because all oxides are soluble in glasses to some extent. The mechanism of the undesirable quality of a metal mold to stick to the glass is the same as that which produces the glass-to-metal seal in the manufacture of light bulbs or radio tubes.

It is very difficult to avoid the sticking of glass to molds, because materials which do not combine chemically with the oxygen ions of the glass are rare. Even gold and platinum have sufficient chemical affinity to adhere to glass at high temperature. In a paper (1) where I discussed this problem, I recommended carburizing or nitriding as a possible means of decreasing adhesion. The most common solution of this problem in the glass industry is the use of carbonaceous materials, wood, graphite, or tar for "lubricating" the mold. These compounds, too, react with the surface of the glass, but the reaction product is a gas (CO, CO_2) and cannot form a bond.

Mr. C. J. Frosch.[3]—Mr. Weyl stated that the electrical surface leakage (insulation resistance) of glass under conditions of high humidity is greatly improved if it is treated with dimethyl silicone dichloride followed by a wash in distilled water. I should like to ask two questions relative to the use of such a treatment in the fabrication of glass cloth laminates which are known to be very poor electrically under conditions of high humidity:

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[1] Director of Research and Development, Federal Telephone and Radio Corp., Newark, N. J.

[2] Coordinator of Technical Information, Owens-Illinois Glass Co., Toledo, Ohio.

[3] Bell Telephone Laboratories, Murray Hill, N. J.

1. Is there any explanation for the failure of this treatment to give an appreciable and permanent improvement in the desirable properties of glass cloth laminates such as insulation resistance and bonding strength under conditions of high humidity or water immersion?

2. Would such a treatment be expected to give a permanent electrical improvement or a temporary one which would disappear in time in the presence of high humidities of free water?

MR. WEYL.—The exposure of a glass surface to water leads to a chemical reaction which is not limited to the surface. Depending on time and temperature of the exposure, water in the form of H^+ and OH^- diffuses into the glass. The "hydrogen glass" conducts the electricity and is responsible for a good part of the surface conductivity. Coating such a glass with dimethyl silicone chloride makes the surface water repellent, but conductivity through the "hydrogen glass" still persists.

I have no experience with the electric properties of glass cloth, but it seems reasonable to expect from its manufacturing process that it contains a considerable amount of "hydrogen glass" which cannot be completely removed before coating.

MR. C. B. HEMMING.[4]—Would Mr. Weyl predict that heat-cleaning of metal in place of customary degreasing and aqueous baths would be satisfactory in producing metal-to-plastic or metal-to-metal bonds of high strength using the new high polymer type adhesives?

MR. WEYL.—It certainly would be worth-while to investigate the behavior of metal surfaces which have been degreased by heat-treating. The process might, however, produce an oxide layer which is too thick and scales off. In the enamel industry, good adhesion is obtained only if the scale is removed by a pickling bath. Heavy metal parts can be sand-blasted.

MR. H. A. EYSENBACH.[5]—Is it possible that the study of adhesion from the approach outlined in this paper would contribute to a solution of the problem which is as follows:

In preparing insulating bushings for the entrance of test leads into a humidity chamber (particularly at saturation), it has been found that adsorbed moisture on the ceramic bushings causes troublesome leakage on the surface of the bushing. In order to overcome this, materials like quartz have been treated with certain silicones to produce surfaces which will not support a continuous moisture film. This treatment has been only partially successful, however, because in many instances the treated material shows low insulation resistance at the interface between the surface coating and the base material. This has been interpreted as evidence of porosity in the surface treatment.

MR. WEYL.—The phenomenon you describe is not necessarily an indication of porosity of the insulator. All silicates, even quartz, react chemically with water to a certain extent. As pointed out in my answer to Mr. Frosch, this reaction leads to H^+ and OH^- ions which produce a certain electric conductivity underneath the water-repellent layer.

MR. E. B. COOPER.[6]—Mr. Weyl stated that adhesion due to van der Waal's forces becomes greatly weakened at temperatures above the point at which rotation of molecular segments begins to take place. Has this loss in adhesion been correlated with other phenomena which are associated with the temperature at which molecular rotation is thought to occur, commonly designated as the second order transition temperature?

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[4] Chief Chemist, United States Plywood Corp., New Rochelle, N. Y.

[5] Engineer, James G. Biddle Co., Philadelphia, Pa.

[6] Physicist, Chemical Division Laboratory, E. I. du Pont de Nemours and Co., Inc., Plastics Department, Arlington, N. J.

MR. WEYL.—I am inclined to think of the van der Waal's forces as only the first steps in the formation of a bond. Once the molecules have been attracted they exert an orienting influence upon each other and some bond will result which is stronger than the van der Waal's attraction. One cannot expect that the molecules which are located at an interface between two media begin to rotate at the same temperature as those in the interior of one of the two phases.

MR. HENRY GRINSFELDER.[7]—Mr. Weyl has stated that the adhesive forces due to dipole moments are greater at room temperature than at elevated temperatures, explaining this by indicating that the rate of rotation of the dipole is slower at the lower temperature and hence the adhesive forces are greater. Why is it then that most adhesives give weaker bonds at temperatures below room temperature, even when the cure is conducted at room temperature?

MR. WEYL.—It is not possible to give a general explanation to this very interesting phenomenon. Theoretically the mechanical strength of matter should decrease with increasing temperature. However, for many substances—glasses and some metals—the tensile strength goes through a maximum value. I made an attempt to explain this phenomenon especially for glasses.[8] The apparent weakening of many substances with decreasing temperature is due to their loss of plasticity. A certain plasticity enables a material under load to dissipate the most dangerous stresses.

MR. G. M. RAPP.[9]—With reference to the phenomenon involved in the crackling of glass by glue, how can the relatively weak organic glues, having tensile strengths probably on the order of 5000 psi. or less, cause stresses that will produce the typical surface chipping characteristic of the process when glass itself has a much higher tensile strength, perhaps on the order of 7000 to 10,000 psi.

MR. WEYL.—For explaining the action of glue on a glass, one must consider the distribution of the forces. A thick layer of the glue might produce stresses which are located only in a very thin layer of the glass.

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[7] The Resinous Products and Chemical Co., Philadelphia, Pa.

[8] W. A. Weyl, "The Mechanical Strength of Glass," *The Glass Industry*, Vol. 27, p. 17 (1946).

[9] John B. Pierce Foundation of Connecticut, New Haven, Conn.

METHODS FOR DETERMINING THE SPECIFIC GRAVITY OF WOOD AND WOOD-BASE MATERIALS*

By L. J. Markwardt[1] and Benson H. Paul[1]

Synopsis

Because of the relation of density to the strength properties of wood and wood-base materials, its evaluation in some terms, such as specific gravity, is an essential requirement for many purposes.

The Standard Methods of Testing Small Clear Specimens of Timber (D 143 – 27)[2] and the Tentative Methods of Testing Plywood, Veneer and Other Wood and Wood-Base Materials (D 805 – 45 T)[3] present methods for determining the specific gravity of wood and wood-base materials for specimens of standard form and size in connection with the determination of strength properties but do not cover methods such as are essential for very large or very small and odd-shaped specimens, or procedures applicable to veneer. This paper presents a summary of the various methods and laboratory techniques that are applicable to all classes of wood and wood-base materials, including measurement, and immersion techniques (both water and mercury), new rapid approximate methods of estimating specific gravity, and a description of apparatus to facilitate evaluation.

The advantages and disadvantages of the several procedures, together with supporting data on accuracy, are presented.

Purpose of Investigation

One of the characteristics of wood is the variation in density (and other properties) among species and within a species that results from heredity and growth conditions. Species of wood differ about 10 to 1 in average density from the heaviest, such as *lignum-vitae*, to the lightest, such as balsa. Individual specimens of a given species may vary as much as about 3 to 1 in density (based on oven-dry weight), including both the variation among different trees, and variations in specimens from different parts of the same tree. The frequency distribution for the specific gravity of individual specimens of a species (random selection) is such that the probable variation is about 8 per cent, based on the mean. Since the specific gravity of wood substance is practically constant for all species (1.53), it is obvious that differences in density result from variations in structure, that is, in the amount of air spaces present, as evidenced by the relative thickness of cell walls, and the relative size and number of vessels and pores (**11**).[4]

These variations in the density of wood provide, on the credit side, material with a range in properties having particular adaptation to many special uses. Because of the relation of the density of dry wood to its other properties, density

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[*] Presented at the Forty-ninth Annual Meeting of the Society, June 24–28, 1946.

[1] Assistant Director and Silviculturist, respectively, Forest Products Laboratory, Madison, Wis.

[2] 1944 Book of A.S.T.M. Standards, Part II, p. 730.

[3] 1945 Supplement to Book of A.S.T.M. Standards, Part II, p. 155.

[4] The boldface numbers in parentheses refer to the references appended to this paper, see p. 1539.

has long been recognized as one of the important properties requiring evaluation. Such determinations are made not only to afford correlation with other properties, but also as a simple and convenient means of selecting wood of desired qualities for special uses.

It is the purpose of this paper to analyze broadly the problem of making specific gravity determinations on wood and wood-base materials, to review such A.S.T.M. methods as have been adopted (D 143 – 27,[2] D 198 – 27,[5] D 805 – 45 T[3]), to present data and suggestions regarding new techniques and developments, and to present a check list of appropriate methods that may be used for samples of any given shape, size, or moisture condition.

DEFINITION OF SPECIFIC GRAVITY

Specific gravity is the ratio of the weight of any volume of a substance to the weight of an equal volume of some substance taken as a standard, usually water. On the basis of water, the specific gravity of wood may range from a fractional part to more than unity. For example, the average specific gravity of white oak (*Quercus alba*) is 0.71 (based on weight and volume when oven-dry) and that of mangrove (*Rhizophora mangle*) is 1.06.

FACTORS INVOLVED

The evaluation of specific gravity, as evidenced by the definition given above, necessitates determining both the weight and volume of the specimen and relating these data to the weight of an equal volume of water. This relatively simple basic procedure, however, encounters a number of complications due to the properties of wood. Wood being hygroscopic, contains moisture in varying degrees which must be considered in connection with the determination of weight; dry wood when placed in water may quickly absorb moisture; the volume is not constant, but changes because wood shrinks and swells with changes in moisture content below the fiber-saturation point; the porous structure, with large vessels open to the surface in some species, places limitations on methods used; and structure and surface conditions sometimes necessitate special consideration in determining volume. An understanding of these factors and their effect on specific gravity is essential to the establishment of techniques and methods.

The principal factors that must be considered in determining density are (*a*) moisture content, (*b*) volume changes, (*c*) porous structure, (*d*) volume measurement methods, especially for thin materials, and (*e*) sampling.

Moisture Content:

Wood has a porous structure. In the living tree the cell cavities, intercellular spaces, and cell walls contain moisture in varying degrees, the average moisture content ranging from 30 to over 200 per cent (based on the oven-dry weight of the wood). It varies among species, in heartwood and sapwood, and in different parts of the tree. Most of this moisture must be driven off in drying wood to make it suitable for the vast majority of uses, yet being hygroscopic, wood used even dry contains some moisture. Wood, if left in an atmosphere at a uniform temperature and relative humidity, will ultimately come to an approximate equilibrium in weight. It is fundamentally important and significant that *the amount of moisture retained ~~at~~ any set of equilibrium conditions is proportional to the dry weight of the specimen, regardless of its density and generally regardless of the species.* For this reason, and because of other correlated relationships, *the moisture content of wood is most generally*

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[5] Standard Methods of Static Tests of Timbers in Structural Sizes, 1944 Book of A.S.T.M. Standards, Part II, p. 761.

expressed as a percentage of the oven-dry weight, and *not as a percentage of the total weight.*

It follows from these facts that the most generally useful and *simplest weight basis for expressing the specific gravity of wood and wood-base materials is that of the oven-dry wood.* Knowing the density based on weight when oven-dry, the weight when moisture is present in any degree can, of course, be readily computed.

It has been shown that, in the living tree, moisture is present in the cell cavities, the intercellular spaces, and the cell walls. In normal drying the first moisture lost is that from intercellular spaces and cell cavities. Not until this "free" moisture has been evaporated does the moisture leave the cell walls. The condition at which no "free" moisture remains, while the cell walls are still fully saturated, is called the "fiber-saturation point." The moisture content at "fiber-saturation" ranges from about 22 to 35 per cent among species. With further loss of moisture below the "fiber-saturation point," namely, with removal of moisture from the cell walls, shrinkage takes place. The amount of shrinkage is closely proportional to the loss of moisture from "fiber saturation" to the oven-dry condition, and varies from a few per cent to as much as 25 per cent among species over this range. Conversely, the amount of swelling of wood in varying stages of dryness depends on the amount of moisture absorbed. The shrinkage of wood takes place principally in the across-the-grain directions, but in normal wood is practically negligible longitudinally. Many wood-base materials are more or less hygroscopic and are subject in varying degree to changes in volume with change in moisture content. Consequently, it is obvious that *any values for specific gravity of wood and wood-base materials must be tied in with the exact moisture condition at which the volume was determined.* Also, that the immersion-in-water method of determining volume cannot be applied accurately to dry material without moisture-proofing the specimen.

Porous Structure:

Because of its porous structure, wood presents certain difficulties when specific gravity methods commonly employed with nonporous materials are used. Since water is commonly used as a standard for comparison in specific determinations, a ready means of determining the volume of a specimen is to obtain a measure in suitable units of the water displaced when it is immersed. The metric system lends itself aptly for the measurement, since the weight of 1 cu. cm. of water equals 1 g., and the determination of specific gravity becomes the simple computation:

$$\text{Specific gravity} = \frac{\text{weight of specimen in grams (in air)}}{\text{volume of specimen in cubic centimeters}}$$

in which the volume numerically in cubic centimeters is the numerical equivalent of grams of displaced water as determined by weighing during immersion.

The porosity of wood, however, offers some complications when the volume is determined by immersion in a liquid.

Wood freshly cut from the living tree will not absorb appreciable quantities of water during a brief immersion to ascertain its water displacement. As soon as any drying of wood has taken place, however, the pores of the wood must be sealed before immersion in water or else the volumetric displacement of the wood specimen will be in error in an amount equal to the volume of water absorbed by the wood. A practical method of preventing moisture absorption of specific gravity specimens is to apply a thin water-resistant surface coating by dipping them into hot paraffin. If all excess paraffin is wiped off before cooling, the volume of the specimen is not materially changed.

In the standard procedure for specific gravity and shrinkage in volume at the Forest Products Laboratory the volume is determined when the specimen is green and again when oven-dry. It is not practical to determine volume by water immersion in intermediate stages of drying for the same samples since the application of a paraffin dip would interfere with the weight of the specimen to be determined later when oven-dry.

Volumes of partly dry samples are most readily determined by measurement after careful cutting to rectangular form or by displacement of a nonabsorptive liquid, such as mercury.

The use of mercury, as an immersion medium, however, is not satisfactory for all wood specimens, since in certain ring-porous woods the mercury enters the large pores and results in an erroneous volume determination. Oak is such a species in which large vessels or ducts show as depressions or cavities on the side and end grain surfaces of a specimen. In the determination of volume, the over-all boundary, exclusive of surface depressions, is obviously that required. Hence, when volume is determined by immersion in mercury in such species, the liquid may penetrate into the pores with resulting error. This being a surface effect, the error obviously varies with the size of specimen and becomes very appreciable for small specimens.

Measurement Methods:

When the volume is determined by measurement of specimens in the form of rectangular parallelepipeds, accurate results can be obtained if the surfaces are smooth and if the dimensions are substantial. The measurement of the thickness of thin veneer presents a difficult problem and special techniques must be used with respect to the anvil size of thickness gages and the pressure employed. As will be shown, reliable determinations of specific gravity of veneer as thin as $\frac{1}{50}$ in. can be obtained with these proper techniques.

Sampling:

Sampling for specific gravity determinations with wood presents a special problem. Since wood varies in density within the cross-section and height of a tree, considerable variation may be found among small samples, say 1 by 1 by 1 in. in size, from a board or a timber, and a single such sample cannot be selected to represent the specimen as a whole. No general procedure is outlined to cover sampling for all requirements. Sometimes density determinations must be made on specimens as small as $\frac{1}{8}$-in. cubes, but usually larger specimens can be chosen.

In the procedure for the systematic evaluation of the properties of wood in accordance with A.S.T.M. Methods D 143 – 27,[2] a density determination is made on each test specimen (except shear, cleavage, and tension) and on six 2 by 2 by 6-in. specimens from each bolt in conjunction with the determination of shrinkage in volume.

Density determinations to evaluate the properties of the wood in living trees have established a practical means of selecting trees to provide wood to meet special needs where the end use justifies the extra cost. Density values representing the quality of material at 1 or 2-in. intervals along a single radius from the bark to the pith have served acceptably for such selection, the samples having been taken with an increment borer.

Basis for Expressing Specific Gravity:

From the analysis of factors involved in the determination of the specific gravity of wood, it follows that any density figure to be precise, and to avoid confusion, must be qualified to establish concomitant weight and volume bases. For example, the average specific gravity of sugar maple (*Acer saccharophorum*) is as follows for three conditions:

0.56—based on weight when oven-dry and volume when green,
0.63—based on weight when oven-dry and volume at 12 per cent moisture content, and
0.68—based on weight and volume when oven-dry.

From such a series of values, it is possible to calculate the percentage change in volume associated with the difference in the density values. Conversely, knowing the average shrinkage of volume, and the shrinkage-moisture relations, it is possible to convert the specific gravity at one condition to that at another. Also such specific gravity figures can be converted to density on a weight per cubic foot basis.

It may be contended that certain density values for wood, such as the specific gravity based on weight when oven-dry and volume at 12 per cent moisture content, represents an artificial condition in that the weight and volume bases are not concomitant. This is, of course, true, but practically, the bases established have proven to be most useful as viewed from long experience.

METHODS OF DETERMINING SPECIFIC GRAVITY

While there is obviously but one procedure for determining the weight in air of specific gravity specimens, there are two basic procedures for determining volume, as follows:

1. Volume by measurement:
   (*a*) specimens in the forms of rectangular parallelepipeds,
   (*b*) samples by taking borings with a Forstner bit, and
   (*c*) samples taken by increment borers.
2. Volume by immersion in liquids:
   (*a*) water, and
   (*b*) mercury.

Measurement:

Rectangular Parallelepipeds.—Determination of volume of specimens in the form of rectangular parallelepipeds can readily be made by measurement, assuming the measurements are made with suitable accuracy. Special care must be used in determining the thickness of thin veneer, however, since an error of 0.001 in. in measuring the thickness of $\frac{1}{50}$-in. thick veneer would cause an error of 5 per cent in the volume determination.

Borings with Forstner Bit.—A convenient method of determining the specific gravity of samples from large units, such as logs or timbers, where it is not practical to saw out a specimen, is by means of borings with a special bit, such as the Forstner type bit which does not have a lead screw. The boring is made to the desired depth, and the volume is established from the diameter and depth measurement.

Increment Borer Samples.—Increment borers, which are designed to take a pencil-sized cylindrical sample from the standing tree, have been employed mainly to determine the growth rate of trees. Such borings can also be used to afford an accurate evaluation of the specific gravity of the wood. With the diameter of the boring accurately established, the length of the sample is measured to determine the volume. The weight of the sample can, of course, also be readily obtained. With increment borer samples, it is also possible to deter- large redwood trees, but the most common form is the small diameter borer.

Immersion in Liquids:

The common liquid-immersion methods usually involve the use of water or mercury to determine the volume. Both can be satisfactorily employed for speci- mine the density of the wood for different distances from the bark to the pith. Some special increment borers as large as 2 in. in diameter were designed and used for selecting density samples from mens of practically any size. Except as limited by errors due to porosity of specimens with large pores, and odd-shaped specimens with long splinters, the mercury has certain advantages for very small specimens. There is at least one special design of a mercury volume meter produced commercially in which the mercury displacement is read on a calibrated scale used to register the movement of a manually operated cylindrical plunger.

TABLE I.—MAXIMUM ERROR OF SPECIFIC GRAVITY DETERMINATION FOR SPECIMENS OF ANY SIZE[a], WHEN WEIGHED AND MEASURED WITH A PRECISION OF ONE-HALF OF 1 PER CENT

	Per cent
Error in same direction:	
Measurement method	±1.0
Liquid-displacement method	0.0
Error in opposite direction:	
Measurement method	±2.0
Liquid-displacement method	±1.0

[a] Calculations based on consideration of specimens ranging from 2 by 2 by 6 in. to 0.1 by 0.25 by 1.0 in. in dimension.

When wood specimens are immersed in mercury, and a balance is used to determine the weight, the balance reaction is always negative, in that the mercury is always much denser than the specimen regardless of species or moisture condition. Some species of wood, both when green and oven-dry, are less dense than water; some when oven-dry are less dense than water, but in the green condition are denser; and others both when green and oven-dry are denser than water.

ACCURACY

From the standpoint of accuracy, the evaluations of density by the basic methods previously discussed, may be classified as (*a*) approximate and as (*b*) accurate.

TABLE II.—SUMMARY OF VARIOUS SPECIFIC GRAVITY METHODS RECOMMENDED FOR WOOD AND WOOD-BASE MATERIALS.

Method	Designation and Application of Method	Literature Cited
1a[a]	Measurement method for large specimens of regular form	...
1b[a]	Water-immersion method for large specimens of regular or irregular form	2
2	Measurement method for large thin plywood panels	8
3a	Measurement method for medium-sized specimens of regular form, (A.S.T.M. Methods D 143 – 27)[b]	[b], 1
3b	Water-immersion method for medium-sized specimens of regular or irregular form (A.S.T.M. Methods D 143 – 27)	[b], 1
4	Buoyancy-immersion method for medium-sized specimens of regular or irregular form	...
5[a]	Flotation-upright tube method for elongated lumber specimens of regular form	5
6[a]	Immersion-graduated tube method for miscellaneous samples	...
7	Mercury balance method for medium- to small-sized specimens	10
8	Measurement method for veneer, plywood, and wood-base materials (A.S.T.M. Methods D 805 – 45 T)[c]	[c]
9	Measurement method for veneer and plywood, Jolly balance	7
10	Measurement method for Forstner bit samples	6
11	Measurement method for increment cores, Jolly balance	6
12	Breuil mercury volumeter for very small samples	4

[a] Approximate methods.

[b] Standard Methods of Testing Small Clear Specimens of Timber, 1944 Book of A.S.T.M. Standards, Part II, p. 730.

[c] Tentative Methods of Testing Plywood, Veneer, and Other Wood and Wood-Base Materials, 1945 Supplement to Book of A.S.T.M. Standards, Part II, p. 155.

Accurate Methods:

Accurate evaluation of specific gravity can be made when volume is determined either by measurement or by the immersion method. The attainment of an accurate evaluation, however, depends also on the necessity of accurately determining moisture content. The most common standard method of determining the moisture content is to find the loss in weight of the specimen when oven-dried to constant weight at 100 C., the moisture content being expressed as a percentage of the oven-dry weight.

Oven-drying of specific gravity and moisture specimens usually requires from one to four days, depending on the size and form, and on the initial moisture content. Furthermore, if the specimen is green and of considerable size (say 2 by 2 by 6 in.), and volume is desired after oven-drying, it should be air-dried for a considerable period, sometimes months, before being placed in the drying oven to avoid case-hardening, checking, and warping. On this account, the accurate evaluation of specific gravity involving the simultaneous determination of moisture content cannot be made instantaneously, but requires one or more days. This time element is the one serious handicap to the application of accurate specific gravity methods to quality control in the wood-using industries, to classification in marketing, and to the selection of wood for special uses.

An analysis of the accuracy attainable by weighing and measuring to one-half of 1 per cent is presented in Table I.

Approximate Methods:

Because of the considerable time required for the accurate evaluation of specific gravity, much study has been given to the development of approximate methods that will permit instantaneous

TABLE III.—CHECK LIST FOR SELECTING THE SPECIFIC GRAVITY TEST METHOD RECOMMENDED FOR SPECIMENS OF DIFFERENT SIZES, SHAPES, AND MOISTURE CONDITIONS.

(Methods are recommended by reference numbers identified in Table II.)

Kind and Size of Specimen	Moisture Condition and Shape of Specimens or Samples					
	Green		Seasoned		Oven-dry	
	Regular	Irregular	Regular	Irregular	Regular	Irregular
WOOD						
Standing trees	10, 11	...	...	...		...
Large timbers	1a, 10, 11	1b	1a, 10, 11	...	...	
Logs, short sections	...	1b	...	...	...	...
Pulpwood	...	1b	...	...	...	...
Medium-sized specimens	3a, 3b	3b	3a	...	3a, 3b	3b
Medium-sized elongated specimens	6	6	5, 6	6	5, 6	6
Small specimens	7, 3a, 3b	7, 3b	7, 3a	7	7, 3b	7, 3b
Very small specimens	12	12	12	12	12	12
VENEER AND PLYWOOD						
Veneer	8, 9	...	8, 9		8, 9	
Plywood, thin large panels	...	...	2	...	2	
Plywood, thin small specimens	...	...	8, 9	...	8, 9	
Plywood, small specimens	...	...	3a, 8	3b	3a, 8	3b
Plywood, very small specimens	...	...	12	12	12	12
MODIFIED WOOD						
Compreg, large panels	...	...	1a, 2	1b	1a, 2	1b
Compreg, medium-sized specimens	...	...	3a, 8	3b	3a, 8	3b
Compreg, small-sized specimens	...	...	3a, 7	3b, 7	3b, 7	3b, 7
Compreg, very small specimens	...	...	12	12	12	12
Staypak, large panels	...	...	1a, 2	1b	1a	1b
Staypak, medium-sized specimens	...	...	3a, 8	3b	3a, 8	3b
Staypak, very small specimens	...	...	12	12	12	12
FIBERBOARD						
Hardboard, large panels	...	...	1a, 2	...	1a, 2	
Hardboard, small specimens	...	...	3a, 7, 8	3b, 7	3a, 7, 8	3b, 7
Hardboard, very small specimens	...	...	12	12	12	12
Insulating board, large panels	...	...	1a, 2	...	1a, 2	...
Insulating board, small specimens	...	...	8		8	...
Insulating board, very small specimens	...	...	12	12	12	12

determination. The instantaneous methods involve an estimate or an approximation of moisture content.

Instantaneous determinations of moisture content within certain ranges can be made by means of electrical moisture meters with reasonable accuracy. Errors by this method on dry material may be within a few per cent.

Another but less accurate method applicable to dry material is to estimate the moisture content. If the storage conditions and length of drying are known, the estimate of moisture content may be accurate enough to permit estimating the specific gravity, based on oven-dry weight and volume at test, with an error of perhaps 5 to 10 per cent.

SUMMARY OF SPECIFIC GRAVITY METHODS

For convenience, a summary of various specific gravity methods which may be used with wood and wood-base materials is presented in Table II, each of which is identified by a method number and an appropriate name or designation. The method number affords a means of identifying the particular method recommended in Table III for determining the specific gravity of specimens of different composition, size, shape, and moisture condition. More complete information on some of the methods may be found by reference to the literature cited in the last column of Table II.

CHECK LIST FOR SELECTING RECOMMENDED METHODS

It has been shown that the specific gravity of wood and wood-base materials may be determined by a variety of methods, and on samples of practically any size and moisture condition. One procedure may be preferable for one set of conditions, another for other conditions. As a guide to suggested practice, there is presented in Table III a check list of procedures recommended for determining the specific gravity of wood and wood-base materials of different size, shape, and moisture condition. To select the recommended procedure, find in the left-hand column of Table III the particular kind of material and size of specimen under consideration. Opposite horizontally will be found the recommended method or methods, designated by number, corresponding to the moisture condition and the shape of specimen involved. Descriptions of methods designated by numbers in Tables II and III are presented below.

DESCRIPTION OF TEST METHODS

1a. Measurement Method for Large Specimens of Regular Form:

The measurement method is particularly applicable to large sawn timbers. The procedure for determining volume and moisture content as described in A.S.T.M. Methods D 198 - 27[5] may be used. The essential steps are as follows:

(*a*) Measure the cross-section and length to ½ per cent.

(*b*) Weigh the specimen.

(*c*) Determine the average moisture content.

To do this accurately it is necessary to cut a representative sample from the timber and determine the moisture content of the sample. The moisture content of the sample is assumed to be that of the timber.

(*d*) From the volume and moisture content as determined, compute the specific gravity, based on weight when oven-dry, and volume at test, in the usual way.

1b. Water-Immersion Method for Large Specimens of Regular or Irregular Form (2):

The water-immersion method is applicable to large specimens of any form including logs, log sections, pulpwood. timbers, compreg, and staypak.

A tank of suitable size to hold the specimens completely immersed is necessary. In the case of any material lighter than water, facilities must be provided for holding the specimens submerged. (Specimens must not be in condition to absorb water readily during immersion.)

The steps in the procedure are:

(*a*) Weigh the specimens.

(*b*) Place specimen or specimens in suitable tank and add sufficient water to fill tank. Remove specimens and determine volume of water left in tank. The difference of the volume of the full tank and the volume of water remaining after removal of the specimens equals the volume of the specimens.

(*c*) Determine average moisture content and compute the oven-dry weight.

(*d*) Compute the specific gravity based on weight when oven-dry and volume at test.

2. *Measurement Method for Large Thin Plywood Panels* (**2, 10**):

(*A*) General Method

- (*a*) Measure the length, width, and thickness to $\frac{1}{2}$ per cent.
- (*b*) Weigh the specimen.
- (*c*) Determine the average moisture content. To do this accurately it is necessary to cut a representative sample from the panel, and determine the moisture content of the sample. (A.S.T.M. Methods D 805 – 45 T).[3] The moisture content of the sample is assumed to be the moisture content of the panel.

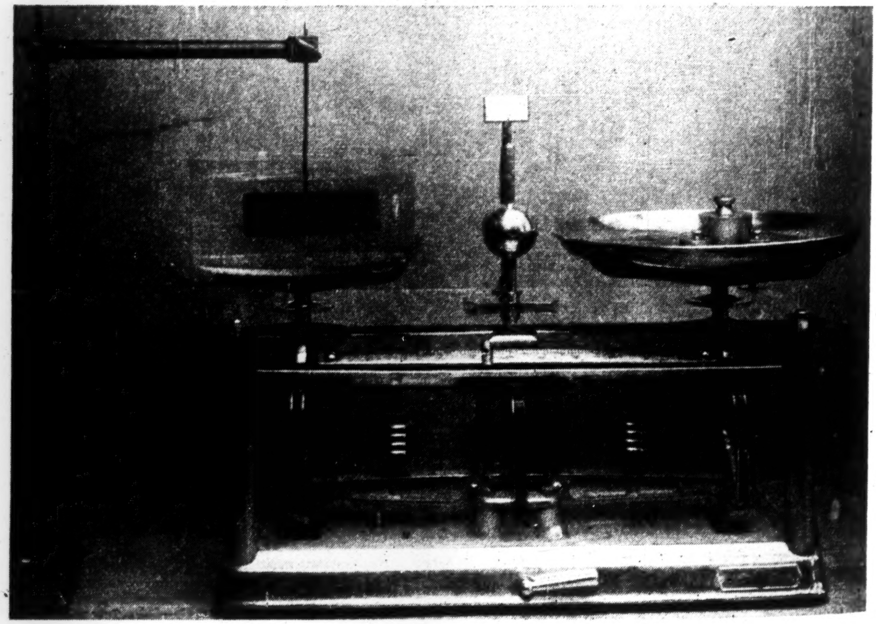

FIG. 1.—Apparatus for Water-Immersion Method of Determining the Specific Gravity of Medium-Sized Specimens (A.S.T.M. Methods D 143).

The container with water shown in the pan on the left is balanced with tare weights placed in the right-hand pan before the specimen is immersed.

(*d*) From the volume and moisture content as determined, compute the specific gravity, based on weight when oven-dry, and volume at test, in the usual way.

(*B*) Special Method for Large Panels that are Oven-Dry or at 10 per cent Moisture Content.

A special method for quickly estimating specific gravity has been worked out for plywood panels that have been uniformly conditioned to a moisture content of 10 per cent (or are known to have this moisture content) and panels that are oven-dry (immediately on removal from press).

A chart is provided from which the specific gravity of plywood panels having a range of thickness and surface areas

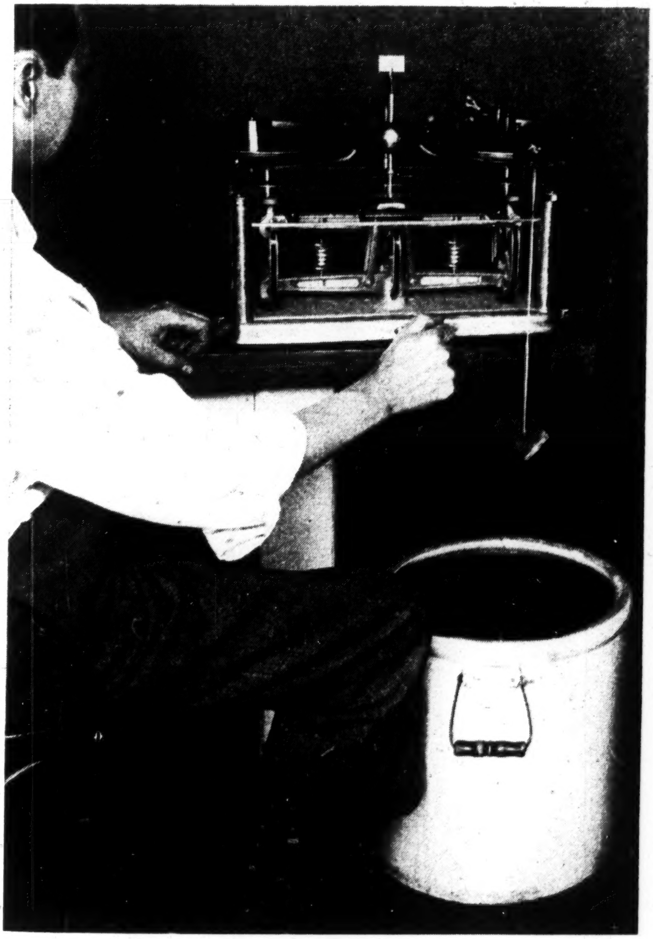

FIG. 2.—Apparatus for Forest Products Laboratory Buoyancy-Immersion Method of Determining the Specific Gravity of Medium-Sized Specimens of Regular or Irregular Form.

The cage for holding the specimen is suspended from the right-hand pan of the balance, and is balanced by tare weights in the left-hand pan. The container for the water may be of any size necessary to accommodate the test specimens.

may be read directly for material at 10 per cent moisture content (also when oven-dry).

3a. Measurement Method for Medium-Sized Specimens of Regular Form (3b):

3b. Water-Immersion Method for Medium-Sized Specimens of Irregular Form:

Methods *3a* and *3b* are described fully in A.S.T.M. Methods D 143 – 27,[2] methods for testing small clear specimens of timber. These methods are applicable to many classes of material, such as compreg, staypak, plywood, and fiber boards. In determining the volume of oven-dry material by immersion in water, the specimen should be dipped in hot paraffin or otherwise given a protective coating to retard absorption of water when immersed. A view of the equipment suitable for use in volume determination by water immersion is shown in Fig. 1.

4. Buoyancy Immersion Method for Medium-Sized Specimens of Regular or Irregular Form:

Method *4* varies from *3b* only in the procedure for obtaining the volume of water displacement by the specimens. Essentially, instead of supporting the specimen rigidly while immersed, the specimen is placed in a cage and immersed in water. The positive or negative buoyancy is weighed on a balance which supports the cage. The cage must be sufficiently heavy to hold specimens immersed that are lighter than water and must serve also to support specimens that are heavier than water.

A convenient system is to suspend the cage by light chains attached to each end of a stick placed across the right-hand pan of the balance. The left-hand pan of the balance is loaded to balance the cage when suspended and immersed without a specimen. The water container may be placed on the floor or on a stand at a convenient height. The balance is conveniently placed over the water container by extending a piece of board beyond the end of the weighing table and placing the balance as shown in Fig. 2. It is essential that there be no change in weight of the specimen from the time of weighing in air until it is weighed in water. Such difference would be a source of error.

The procedure is as follows:

(*a*) Weigh the specimen.

(*b*) Determine the moisture content.

(*c*) If necessary, moisture-proof the specimen. (A recommended method is by dipping in hot paraffin.)

(*d*) Weigh the specimen after moisture proofing.

(*e*) Load the left-hand pan of the balance to balance the scale when the cage is freely immersed.

(*f*) Place the specimen in or under the cage and submerge in water. Determine the weight necessary to rebalance, by placing weights on the right-hand pan (if the specimen is lighter than water), using the slide of the beam as necessary.

(*g*) The weight in grams added (if the specimen is lighter than water) to the weight of the specimen in air equals numerically the volume of the specimen in cubic centimeters.

(*h*) From the volume and moisture content as determined, compute the specific gravity, based on weight when oven-dry and volume at test.

If the specimens sink in water, as is frequently the case with green wood, the specimen is supported on top of the cage during immersion, if an open cage is used. The weights to rebalance are then placed on the left-hand pan. The weight to rebalance is recorded as before, but in this case it is subtracted from the weight in air to give the volume of the specimen.

This method involves an extra weighing in the case of dry material that is paraffin coated before immersion. The weight in air and the weight when paraffined must both be recorded. The weight to rebalance when the specimen is immersed after being paraffined is added to the weight in air after paraffining to obtain the volume when dry. With common American species of wood, no dry pieces are heavy enough to sink in water.

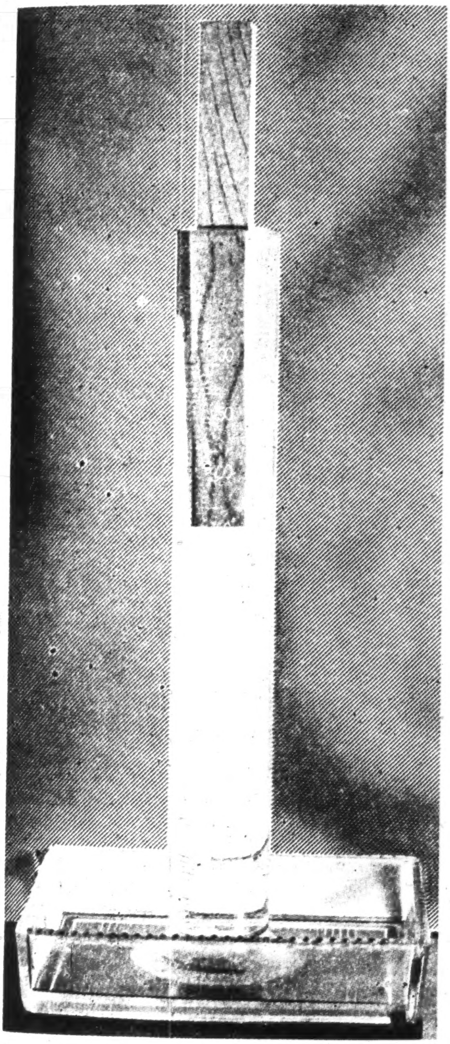

FIG. 3.—Cylinder and Specimen as Used in Flotation-Upright Tube Method for Determining the Specific Gravity of Elongated Lumber Specimens of Regular Form.

This is an approximate method for rapid testing. The cylinder should be filled to capacity with water, and the water line marked on the specimen immediately after immersion.

The advantages of this method are (1) that the water and container do not have to be rebalanced after the immersion of each specimen; (2) that the water is all below the table top and if spilled does not get on record cards or otherwise mess things up; and (3) the method is rapid. With a little practice the volume of specimens may be secured at the rate of one per minute. The most confusion exists when a portion of the specimens float and others sink in water, but this offers no hindrance if the system is thoroughly understood, or time may possibly be saved by first dividing the pieces that sink from those that float.

5. *Flotation Upright Tube Method for Elongated Lumber Specimens of Regular Form* (7):

A quick method for determining the comparative specific gravity of dry pieces of wood that have the same moisture content consists of floating elongated rectangular specimens with parallel sides in a tube containing water (Fig. 3). A convenient length of specimen is 10 in., and a convenient cross-sectional area is approximately 1 sq. in. However, other lengths and cross-sections may be used.

If oven-dry specimens exactly 10 in. in length are used, the computation of specific gravity consists merely in dividing the submerged length in inches by 10. If specimens are not oven-dry, but of a known moisture content, corrections may be made to reduce the reading to an oven-dry value by use of an appropriate constant.

In production control where segregation of the very light or the very heavy material is desired, and the moisture content among specimens is uniform, the allowed immersion range corresponding to the control limits may be marked on the specimens before floating. Specimens representing unsatisfactory material may thus be segregated as the testing proceeds. This method is particularly applicable where it is desired to group the wood of a species in a small number of density grades.

6. *Immersion-Graduated Tube Method for Miscellaneous Specimens:*

A rapid approximate method consists of immersing an elongated specimen in a graduated tube of a cross-section only slightly larger than that of the specimen. This insures a relatively large movement of the water level, which can be read directly from the graduations before and after immersion (Fig. 4). The method, like others, requires a knowledge of the moisture content of the material. To evaluate moisture content, the specimens may be oven-dried or a moisture-content determination may be made. Another suggestion, where facilities are available, is to condition the specimens to equilibrium in a temperature-humidity controlled room, bringing them to some desired moisture condition. If the specimens are oven-dried, it is preferable to moisture-proof them by dipping in hot paraffin to secure most accurate results.

The procedure is as follows:

(*a*) Prepare elongated specimens, such as a strip from the edge or end of a board, and weigh the specimen.

(*b*) Determine the moisture content.

(*c*) Place water in a graduated flask or cylinder, preferably filling to an even graduation mark, such as 100 cu. cm. and read the water level.

(*d*) Place the specimen in the flask, holding it immersed with a pointer if necessary and read instantly the water level. It is necessary to perform this operation quickly to determine the water level before moisture can be absorbed by the specimen unless it is paraffin coated.

(*e*) If the graduations of the flask are in cubic centimeters, the difference between before and after water-level readings equals the volume of the specimen in cubic centimeters.

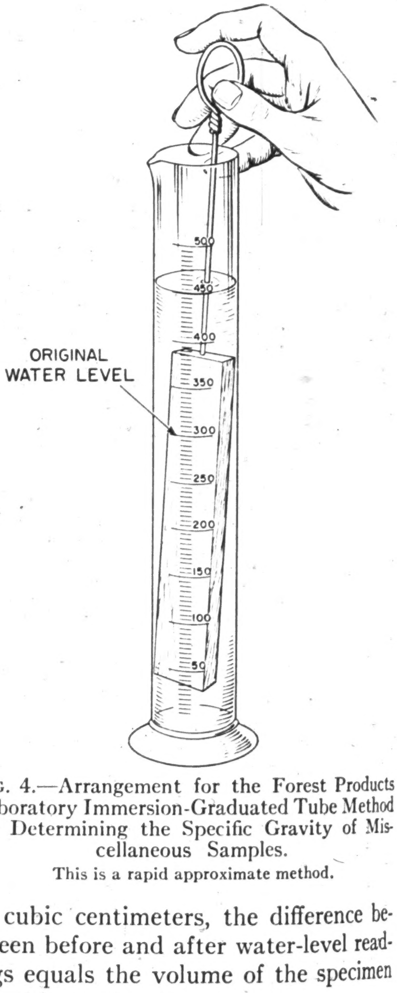

FIG. 4.—Arrangement for the Forest Products Laboratory Immersion-Graduated Tube Method for Determining the Specific Gravity of Miscellaneous Samples.
This is a rapid approximate method.

(*f*) From the volume and moisture content as determined, compute the specific gravity, based on weight when oven-dry and volume at test.

7. *Mercury Balance Method for Medium to Small-Sized Specimens* (10):

The mercury balance method, described in detail by Stern and Dear

(10),[6] is applicable to medium and small-sized specimens. In this test, the specimen is held submerged in mercury by a rigid attachment to the weighing balance. The volume is computed from the weight of the specimen in air and the "weight" in mercury, using the appropriate conversion constant determined for the particular balance equipped with the necessary mercury-immersion devices.

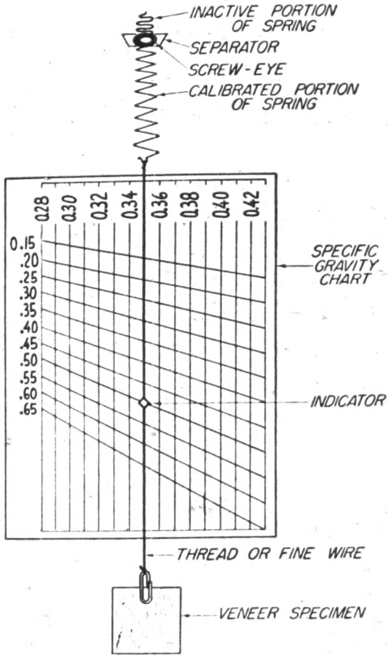

FIG. 5.—Apparatus Employing a Jolly Balance Used in the Forest Products Laboratory Measurement Method for Determining the Specific Gravity of Veneer and Plywood.

This is a rapid method applicable to inspection use. A special chart and indicator permits the direct reading of the specific gravity.

8. A.S.T.M. Measurement Method[3] *for Veneer, Plywood, and Wood-Base Materials:*

In conjunction with the testing of plywood, veneer, and other wood and wood-base materials, provision is made for the determination of specific gravity of the test material, either on the test specimen itself, or on a sample therefrom. The same specific gravity methods may in general be used when density alone is desired.

The general procedure for specific gravity determinations on these materials is as follows:

(*a*) Weigh and measure the specimens.

(*b*) Determine the moisture content, using either the entire specimen for a moisture sample, or a small sample cut from it. With resin-treated wood, such as compreg, the moisture content is relatively low, and the volatile material driven off after 240-hr. drying in an oven at 100 F. will range from 3 to 5 per cent. With such material, the density determination is not materially affected by moisture content, as in the case of wood.

(*c*) From the data obtained, compute the specific gravity in the usual way.

9. Measurement Method for Veneer and Plywood; Jolly Balance (7):

A rapid production method for determining the specific gravity of small veneer specimens makes use of a sensitive helical spring of the type used in a Jolly balance, to which is attached a light cradle for supporting the specimen. A standard specimen of fixed length and width is employed (cut out with a trimmer), and the thickness is measured. Charts have been prepared for specimens 75 sq. cm. in area, for thicknesses ranging from 0.38 to 0.58 in., and for specimens of 100 sq. cm. in area for thicknesses ranging from 0.28 to 0.43 in. An indicator is attached to the lower end of the spring. When the thickness of the specimen is known, the specific gravity is read directly from the indicator on a special chart attached to the apparatus (Fig. 5).

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[6] E. George Stern and Paul S. Dear, "A Simplified Method for the Determination of the Specific Gravity of Wood and Plastics," A.S.T.M. BULLETIN No. 135, August, 1945, p. 35.

Individual Jolly balance springs are readily adjusted to the chart simply by altering the point of suspension, thus changing the effective length of the spring.

The steps in determining the specific gravity, after the apparatus is adjusted, are as follows:

(*a*) Cut the veneer specimens to the desired surface area, using a cutter provided with a stop to assure accurate dimensions. If specific gravity based on weight and volume when oven-dry is desired, the specimens must be oven-dried before cutting to exact size.

(*b*) With a thickness gage of the dial type, determine the average thickness of specimen to the nearest 0.001 in. The suggested procedure is to caliper near each corner and take the average of the four measurements. The contact surfaces of the micrometer should be equipped with slightly rounded anvils to insure accurate results, as this is an important factor in the precision of the method.

(*c*) Determine the moisture content of the veneer. The procedure with respect to moisture content depends on the purpose. If the specimens are oven-dried, accurate results are obtainable for specific gravity, based on weight and volume when oven-dry. If the moisture content is estimated, the method is only approximate, the accuracy depending on the accuracy of the moisture estimate. However, in many instances the moisture content may be estimated within a few per cent.

(*d*) Suspend the specimen from the spring on the thread passing in front of chart, slide the chart so that the line corresponding to the thickness of the specimen is directly back of the thread, and read the specific gravity directly as shown by the indicator. The value is that for specific gravity based on weight and volume when oven-dry.

(*e*) If the specimens are not oven-dry, suitable correction for moisture must be made.

Care should be used in preparing specimens to avoid rough edges. The measurement of thickness is important. A dial micrometer having anvils with rounded surfaces has been found to give consistently accurate results. It is possible that such a micrometer provided with a constant pressure weight would prove even more satisfactory. For rapid production, however, the method as described has proved sufficiently accurate.

A sequel to this method (9) provides a formula for computing the specific gravity of plywood when the specific gravity of the constituent plies is known.

10. Measurement Method for Forstner Bit Samples:

The specific gravity of large units, such as logs or timbers, either for selection at source or for determinations on structures in place may be made on borings taken with a Forstner bit. A convenient size is a bit of 1-in. diameter, and a depth of boring of 2 in. The method consists of collecting all the chips from a given boring and basing the determination on the weight of the chips and the volume of the hole. The volume is computed from the diameter of the hole and its depth. A depth limit gage in the case of timbers or flat exterior surfaces can be attached to the bit to control the depth of the boring automatically and yield samples of uniform size. If the hole is bored normal to a vertical surface, the chips can readily be collected by attaching a container such as a cardboard box to the timber underneath the hole by means of thumb tacks. The chips can then be emptied into a small paper bag for convenience in completing the determination.

If the moisture content is desired, the chips must be weighed immediately, and subsequently after drying. If specific gravity only is desired, the chips need be weighed only after oven-drying. The specific gravity obtained is that based on weight when oven-dry and volume at test.

In the use of paper bags for handling the chips, the top of the bags should be left open when placed in the oven for drying the sample. The weight of the bag must, of course, be determined both before and after oven-drying, and used to correct the tare weight of sample to net weight in the calculation of specific gravity. When many determinations are being made, the weight of the individual bag can be taken as the average of a large number weighed at one time. The steps involved are as follows:

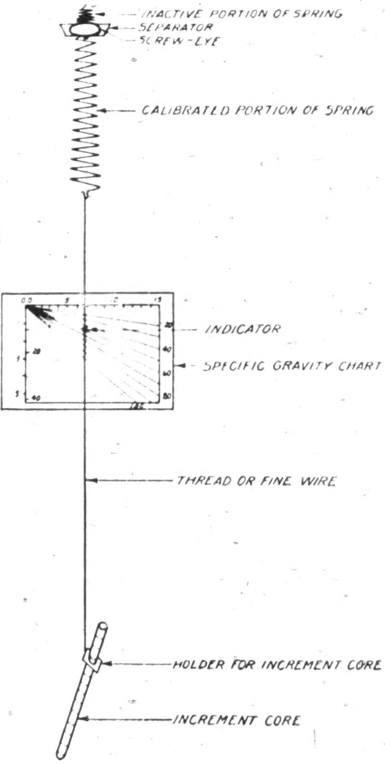

Fig. 6.—Special Apparatus Employing a Jolly Balance for the Forest Products Laboratory Method of Determining the Specific Gravity of Increment Cores.

A special chart and indicator permit the direct reading of the specific gravity.

(*a*) With a Forstner bit, bore a hole normal to the surface of the timber, and collect all the chips.

(*b*) If moisture is desired, weigh the chips immediately.

(*c*) Determine the size of the hole. The diameter may be measured to the nearest 0.01 in., or may be taken as the diameter of the cutting edge of the bit. The depth of the hole should be measured to the nearest 0.01 in. If the diameter of bit is determined and a limit gage is used to control the depth of hole, the volume determination may be standardized for quick operation.

(*d*) Oven-dry the chips, and determine the oven-dry weight.

(*e*) If paper bags are used for retaining the chips, correct the tare weight for the weight of the bag to obtain net weight.

(*f*) From the data obtained, compute the specific gravity, based on weight when oven-dry and volume at test, in the usual way.

11. Measurement Method for Increment Cores, Jolly Balance (6):

Specific gravity determinations can be readily made by means of increment cores taken with a special bit from standing trees, logs, poles, piling, and timbers. The pencil-like cores, after oven-drying, are suspended from a Jolly balance spring, and the specific gravity may be read directly from an indicator in conjunction with a specific gravity chart attached to the apparatus. Such charts may be constructed for use with cores of various lengths, although it is preferable to cut the cores to a standard length, thus simplifying considerably the determination of specific gravity values (Fig. 6).

The volume of the specimens may be computed from the established diameter of the cutting edge of the increment borer and the measured length of the core taken immediately after extraction from trees or timbers and before any shrinkage in length has taken place due to drying. If the moisture content of the specimen is desired, cores must be weighed as soon as extracted. Customarily the specific gravity obtained by this method is that based on weight when oven-dry and volume at test. For fairly dry material of known moisture content, the specific gravity based on weight and volume at test can be adjusted to specific gravity based on weight and volume when oven-dry by using a constant applicable to the species.

With increment cores, the critical measurement is the diameter of the core. This must be determined to the nearest 0.001 in. Since increment borers often vary in diameter by more than this amount, it is a good plan to provide a calibration index for a range of increment borer diameters when constructing the chart.

The steps involved, in addition to the determination of the diameter of the cutting edges of the increment borer and the construction of the chart are as follows:

(*a*) Measure the length of the increment core to the nearest 0.01 in.

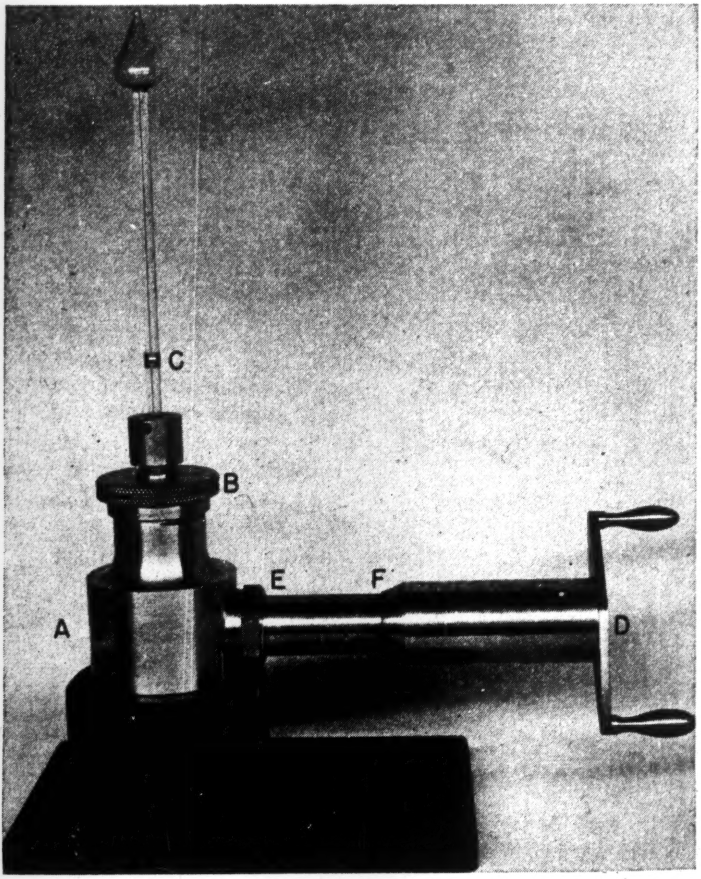

FIG. 7.—Breuil Mercury Volumeter, Consisting Essentially of a Chamber in Which the Specimen Is Immersed, and a Graduated Piston and Cylinder for Measuring the Displacement.

This method is applicable to very small specimens of wood and wood-base materials without large pores.

(*b*) If the moisture content is desired, weigh the core immediately or wrap in vaporproof foil covering to protect the core from moisture changes pending later weighing.

(*c*) Oven-dry the increment cores to constant weight at 100 C. (A small portable insulated drying oven, having over-all dimensions of 4 by 4 by 15 in., and which may be attached to standard electric outlets has been devised for field use for oven-drying cores.)

(*d*) Immediately after removal from the oven, suspend the dried core from the Jolly balance spring in front of the appropriate chart for cores of the length chosen. Then by means of the indicator read the specific gravity of the specimen directly from the chart. This method has been found to be useful in the field for selecting trees having wood of a definite specific gravity. The specific gravity obtained is that based on weight when oven-dry and volume at test.

12. Brueil Mercury Volumeter for Very Small Specimens (4):

A special volume meter has been designated for determining the volume of small specimens of wood and wood-base materials. It consists essentially of a mercury chamber connected with a graduated plunger operating in a close-fitting cylinder. The volume is determined from the difference in the graduated scale readings taken when the specimen is in the instrument and after its removal. The apparatus is shown in Fig. 7. The procedure of operation is as follows:

(*a*) Weigh the specimen to the nearest 0.001 g.

(*b*) If moisture content is desired, oven-dry the specimen, and determine the oven-dry weight.

(*c*) Remove cap *B* (Fig. 7) and place the specimen in the mercury chamber *A* which contains only enough mercury so that when the specimen is submerged the mercury does not overflow. Replace the cap and revolve piston *D* until the mercury is forced up into the capillary tube to a height corresponding to the arbitrarily placed sliding marker *C*.

(*d*) Read the scale on top of the barrel *E* and the vernier *F*.

(*e*) Retract the piston by revolving in the opposite direction until the mercury is below the top of the cap. Then remove the specimen.

(*f*) Replace the cap, advance the piston sufficiently to bring the mercury back to the level of the marker *C* and again read the scale. The two readings are subtracted and the difference is multiplied by 0.3 to give the volume of the specimen in cubic centimeters. The volume is determined to 0.003 cu. cm.

It is obvious that when specimens of woods with large pores are immersed in mercury, some of the mercury will enter the pores and thus affect the specific gravity determination. The error resulting from this cause will be greatest for very small specimens. The most accurate results are obtained with the volumeter with specimens having very small pores. This factor should be kept in mind.

DISCUSSION OF METHODS

Extensive data on the specific gravity of wood obtained in conjunction with the evaluation of properties of different species under A.S.T.M. Methods D 143[2] are available, as well as that obtained in conjunction with other research studies. Of particular interest may be the results of a series of special tests to compare some of the newer methods employing very small samples with the method of D 143.

Comparative results of a series of determinations on mahogany and khaya using (1) the measurement method for increment cores, (2) the measurement method for veneer and plywood, Jolly balance, and (3) the method for medium-sized specimens, A.S.T.M. Methods D 143, all measured on matched samples from the same flitches, are presented in Table IV. Presented also are standard deviation values for the results by the different methods. It is interesting to note the substantial agreement among the average results for the three methods for both species of wood, and among the standard deviation values. Statistically, the results show no significant difference among the methods.

Limitations of space preclude the presentation of the results of the individual tests and data for other methods.

CONCLUSIONS

It has been shown that there are many methods for determining the specific gravity of wood and wood-base materials applicable to specimens of different size, shape, and moisture condition, and to serve different needs. Some have a long record of satisfactory use, but even the newer methods have been applied sufficiently to demonstrate their utility and accuracy. From the methods described and from comparative experimental results obtained, it is evident that the accuracy of results depends upon selection of an appropriate method entailing the necessary degree of precision for the specimens used.

Presented in Table II is a check list for determining the specific gravity test method recommended for specimens of different sizes, shapes, and moisture conditions. It is recognized that for any specific problem several methods may be employed, and it is not possible

TABLE IV.—COMPARISON OF SPECIFIC GRAVITY VALUES AS DETERMINED BY DIFFERENT METHODS.

Species	Method	Type of Specimen, and Size, in.	Number of Tests	Mean Specific Gravity	Mean Difference (Algebraic)	Standard Deviation	Standard Error of Mean Difference	t	Significance of Difference
SPECIFIC GRAVITY BASED ON WEIGHT AND VOLUME AT 10 PER CENT MOISTURE CONTENT									
Mahogany	No. 9[a] No. 3[b]	Veneer 2 x 2 x 6	96 96	0.545 0.552	0.007	0.047 0.045	0.0067	1.045	Not significant
Mahogany	No. 11[c]	Cores 2 x 2 x 6	50 50	0.548 0.560	0.012	0.046 0.047	0.0093	1.290	Not significant
Khaya	No. 9[a] No. 3[b]	Veneer 2 x 2 x 6	95 95	0.502 0.498	0.004	0.041 0.044	0.0062	0.645	Not significant
Khaya	No. 11[c] No. 3[b]	Cores 2 x 2 x 6	72 72	0.486 0.491	0.005	0.044 0.044	0.0074	0.676	Not significant
SPECIFIC GRAVITY BASED ON WEIGHT AND VOLUME WHEN OVEN DRY									
Mahogany	No. 9[a] No. 3[b]	Veneer 2 x 2 x 6	96 96	0.509 0.520	0.011	0.045 0.043	0.0063	1.746	Not significant
Khaya	No. 9[a] No. 3[b]	Veneer 2 x 2 x 6	95 95	0.467 0.470	0.003	0.039 0.042	0.0059	0.508	Not significant
SPECIFIC GRAVITY BASED ON WEIGHT WHEN OVEN-DRY AND VOLUME AT 10 PER CENT MOISTURE CONTENT									
Mahogany	No. 11[c] No. 3[b]	Cores 2 x 2 x 6	50 50	0.494 0.502	0.008	0.039 0.042	0.0081	0.988	Not significant
Khaya	No. 11[c] No. 3[b]	Cores 2 x 2 x 6	72 72	0.443 0.441	0.002	0.039 0.040	0.0065	0.308	Not significant

[a] Method 9, measurement method for veneer and plywood, Jolly balance.
[b] Method 3, method for medium-sized specimens (A.S.T.M. Methods D 143).[2]
[c] Method 11, measurement method for increment cores.

categorically to establish any single specific method.

Because of the many factors involved, such as volume change and moisture content, the determination of specific gravity has many complications. It is not the thought that the procedures described and presented form a complete catalog of all possible methods; rather, the presentation should serve to provide acquaintance with the factors involved, to give detailed procedure for certain definite methods, and to provide a basis for developing new methods or combinations which will most satisfactorily meet any given problem from the standpoint of accuracy, or speed of operation.

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THE COMPREHENSIVE LABORATORY TESTING OF INSTRUMENT LUBRICANTS* [1]

BY G. E. BARKER,[2] G. E. ALTER, JR.,[2] C. E. MCKNIGHT,[2] J. R. MCKLVEEN,[2] AND D. M. HOOD[2]

THE object of this paper is to describe the various laboratory tests employed during the investigation and development of new synthetic lubricants for fine instruments—a comprehensive research conducted during the past three years. These test procedures have been found extremely helpful in accelerating the preliminary determination of the value of oils for these specialized purposes. Of even greater importance is the fact that these laboratory determinations correlate very well with operational experiences.

It has been found that the following properties, which are among the most essential characteristics of a good instrument lubricant, are readily measured in the laboratory:

1. Viscosity,
2. Cloud and pour points,
3. Oxidation stability and corrosion of brass,
4. Corrosion and rusting of steel,
5. Evaporation,
6. Spreading, and
7. Coefficient of friction of steel on sapphire, lubricated.

In selecting, adapting, or evolving these tests two important conditions have been constantly emphasized. First, an effort has been made to keep small the volume of sample. If conducted in the proper sequence, the seven tests listed can be performed on a 1-oz. specimen. This fact is quite important, for instrument lubricants are always expensive and sometimes scarce. Secondly, careful consideration has been given to the simplicity of apparatus and procedure. Specialized equipment, other than that available in any well-equipped chemical laboratory, has been avoided wherever possible. For both evaporation measurement and coefficient of friction determination, however, special apparatus was designed and constructed because no adequate equipment could be purchased.

VISCOSITY

The viscosity is determined by A.S.T.M. Tentative Method of Test for Kinematic Viscosity (D 445 - 42 T),[3] using the modified Ostwald viscosimeters. The viscosities are routinely measured at 100, 32, and --40 F. Occasionally special determinations are made at −60 or −80 F. The low temperatures are obtained by placing a thermostatically controlled isopropanol bath in a low-temperature cabinet maintained about 5 F. below the desired bath temperature.

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[1] Published by permission of the Navy Department.

[2] Multiple fellowship on special lubricants sustained at Mellon Institute by the Instruments Branch, Bureau of Aeronautics of the U. S. Navy Department. On this program see *Chemical and Engineering News*, Vol. 23, p. 524 (1945).

[3] 1944 Book of A.S.T.M. Standards, Part III, p. 1294.

Cloud and Pour Points

The cloud and pour points are found by a modification of the A.S.-T.M. Method of Test for Cloud and Pour Points (D 97 – 39),[4] in which a 16-mm. outside diameter test tube is substituted for the test jar, making it possible to run the tests on a 10-g. sample. In general, the cooling procedure of the standard method is followed; but, as most useful synthetic lubricants have very low cloud and pour points, the test tube containing the sample of lubricant and the thermometer is usually immersed directly in the cooling medium, which may be a mixture of solid carbon dioxide and any low-melting liquid (isopropanol, acetone, etc.).

Oxidation Stability and Brass Corrosion

In the preliminary evaluation of instrument lubricants there necessarily must be provision for determining the susceptibility to oxidation and the tendency to corrosion of the oils under examination. This need is particularly true in the case of synthetic lubricating materials for which there may be available no knowledge upon which to base an estimate of the effect of new compositions and possible impurities upon the properties of the lubricants.

Many of the methods set forth in the literature for determining the oxidation and corrosion properties of oils, either separately or together, have been designed especially for lubricants to be used in internal-combustion engines, and these methods, logically enough, attempt to duplicate the conditions encountered in such engines. None of these tests, therefore, was regarded as particularly well adapted to the examination of lubricants for fine mechanisms. There was required a rapid and simple test that would give some indication of the stability of the materials in the presence of atmospheric oxygen, catalyzing metallic surfaces, and traces of moisture. Important also was some means of determining quantitatively the effect of the oil itself or of any possible oxidation products upon carefully polished metal surfaces such as are found in instruments. Finally, it seemed essential to be able to obtain this information from very small samples. The test method described below was developed in an attempt to combine these requirements into one simple procedure that would enable a rapid and fairly indicative evaluation of the lubricants under study.

Preliminary Considerations:

There were several factors to which attention was given in setting the conditions of the test. First, it was regarded as essential that the oil sample be completely saturated with oxygen during the course of the test, for in some air-driven instruments, such as aircraft gyro instruments, there is prolonged contact between the lubricant and large quantities of atmospheric oxygen. Secondly, it was deemed advisable to use brass as the metallic material in the test because brass acts as an oxidation catalyst, is subject to corrosion by many organic materials, and is one of the metals commonly found in parts of small mechanisms.

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[4] 1944 Book of A.S.T.M. Standards, Part III, p. 115.

Finally, it seemed necessary to include traces of moisture in the test medium in order to simulate to some extent the conditions encountered in service in moist or humid climates.

Procedure:

The method consists of bubbling moist oxygen at atmospheric pressure through a sample of lubricant in contact with a polished brass surface. In order to accelerate the process and to reduce the time required, the test is run at 100 C. (±0.5 C.). The temperature is maintained constant by immersing the sample tubes in an electrically heated and thermostatically controlled oil bath (Fig. 1). Previous to its introduction into the sample, the oxygen is mixed with water vapor by being bubbled through a bottle containing distilled water at room temperature. The moist oxygen is then led down through the oil bath by means of a small tube that makes provision for pre-heating the gas, and is finally admitted to the bottom of the sample tube. A highly polished block of brass is contained in the tube in contact with the oil, and the moist oxygen is caused to bubble up past this block, thus bringing into intimate contact the sample of oil, the brass surface, and the mixture of oxygen and water vapor. Agitation is supplied by the bubbling action of the rising gas. Dust, atmospheric impurities, and oil vapor from the heating bath are prevented from entering the sample tube by inserting in the mouth of the tube a cork stopper that has had a narrow V-shaped groove cut along its length. The groove provides for escape of used oxygen to the atmosphere, but causes sufficient increase in the velocity of the escaping gas to prevent any vapors or particles from entering against the gas stream.

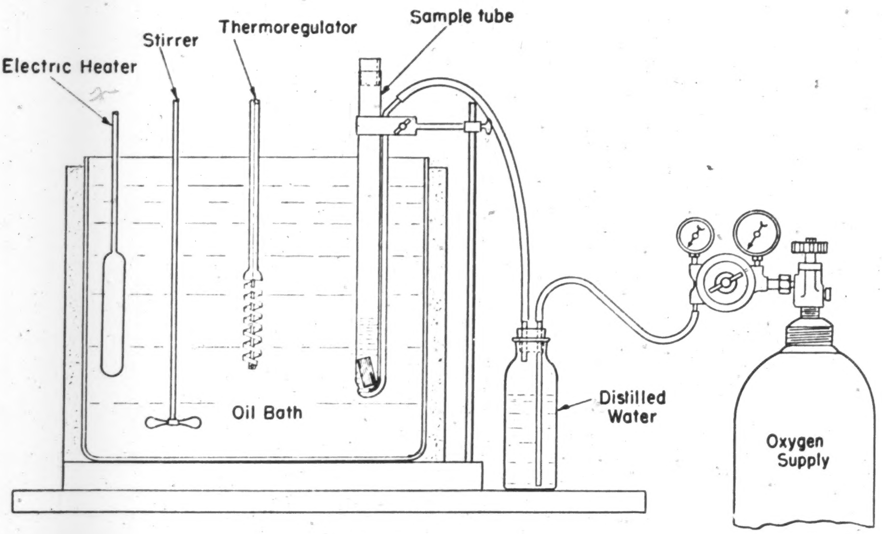

FIG. 1.—Apparatus for Determining Oxidation Stability and Brass Corrosion.

The procedure followed in running the oxidation-stability and brass-corrosion test is as follows:

A small brass block, approximately 1 cm. square and 2.5 cm. long, is cleaned and polished with a series of metallographic emery papers, finishing with the No. 0000 grade. The block is then washed in a stream of water, swabbed with a piece of wet absorbent cotton, and dipped several times into a beaker containing distilled water. From the distilled water the block is transferred by means of a small pair of forceps to a beaker containing ethyl alcohol (95 per cent) and thence to a beaker of benzene. Finally, the block is dried in an oven at 105 C. for 30 min., after which it is cooled in a desiccator.

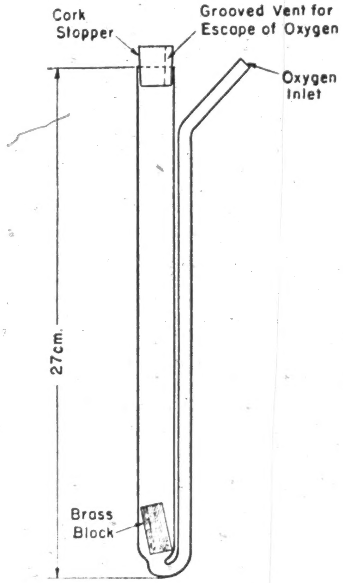

FIG. 2.—Tube Used in Oxidation and Corrosion Test.

After the brass block has been polished and cleaned, it is carefully weighed on an analytical balance and then placed in the bottom of one of the special test tubes (Fig. 2). Subsequently there is added 10 ml. of the oil to be tested, the viscosity of which has been determined at 100 F. (37.78 C.). A cork stopper, grooved as described, is fitted into the open end of the tube, and the tube itself is clamped in a vertical position with at least 18 cm. of its length immersed in the thermostatically controlled oil bath. A small neoprene tube connected to the moist oxygen supply is attached to the gas inlet tube, and the oxygen flow is so regulated that two or three bubbles per second (about 0.2 cu. ft. per hr.) rise through the sample of oil being tested. With the exception of periodic checks on the rate of oxygen flow, no further attention is required for the duration of the test.

At the end of 100 hr., the neoprene tube supplying oxygen is disconnected and the test tube is removed from the oil bath. After the outside of the tube has been cleaned and dried, the lubricant sample is carefully poured from the test tube into a small bottle in which it is kept pending a determination of its viscosity. The brass block is then removed from the tube and placed in a small beaker of benzene. As before, the block is handled only by means of a pair of forceps. After having been rinsed by dipping several times in the benzene, the block is transferred to a beaker of alcohol, where the dipping process is repeated. Finally the block is dried in an oven at 105 C. for $1/2$ hr. and is cooled in a desiccator. Then, as prior to the test, the brass sample is carefully weighed, and the weight found after the test is compared with that previously determined. Likewise, the viscosity of the oil at 100 F. (37.78 C.) after the test is compared with the viscosity of the untested lubricant.

TABLE I.—CORROSION OF BRASS BY LUBRICANTS.

Time of test, 100 hr., temperature, 212 F.

Lubricant	Weight of Brass Block		Change in Weight, g.	Viscosity of Oil at 100 F.		Appearance of Oil After Test	Appearance of Brass Block After Test
	After Test, g.	Before Test, g.		Before Test, centistokes	After Test, centistokes		
Lubricant A (mineral oil, inhibited)	17.7626	17.7626	0.0000	16.2	16.4	Somewhat darkened in color; otherwise unaffected	Unaffected
Lubricant B (synthetic)	18.3305	18.3305	0.0000	11.5	11.7	Slightly darkened, but still clear and without any trace of sludge or sediment	Slightly colored by an irridescent stain, but otherwise unaffected
Lubricant C (mineral oil, inhibited)	19.0009	18.9940	+0.0069	10.3	11.3	Very dark. Considerable amount of sludge	Covered by a white deposit under which the surface was slightly pitted
Lubricant D (synthetic)	18.7613	18.8366	−0.0753	8.8	Undetermined	Dark green, opaque, and containing a large amount of sludge	Badly corroded and covered by a scaly surface film

Examination of Results:

It has been found by experience that oils not unduly subject to oxidation and with little tendency to corrode brass never undergo more than a 5 per cent viscosity change during the test and never cause the brass block to lose more than 1 mg. in weight. These figures, therefore, have been set up in this laboratory as the standards to be met by a lubricant being subjected to the test. But practically any oil sample passing the test is able to meet these standards by a considerable margin, and, conversely, a sample that fails usually does so very definitely. In Table I are summarized the results obtained on four oil samples, of which two passed the test and two failed.

Conclusion:

It will be noticed that this test is in many respects similar to the original Indiana oxidation test (**1**)[5] for motor oils. The fundamental differences lie in the temperature to which the oil sample is heated and in the method by which the results are evaluated. It did not seem necessary to subject instrument lubricants to the high (172.2 C.) temperature used in the Indiana test to obtain correlations with conditions in the crankcase of an internal-combustion engine. Neither did it appear advisable, in view of the frequently small samples available, to try to measure the time required to reach some predetermined end point by periodically extracting and examining portions of the oil sample being tested. The advantages believed to be inherent in the present procedure for testing instrument lubricants are as follows:

1. The test provides, under conditions which are not too radically different from those which might be encountered in service, a fairly rapid method of measuring the extent to which an oil is susceptible to oxidation.
2. The test gives an indication of the degree to which either the oil or its possible oxidation products are likely to affect the polished metal surfaces found in fine mechanisms.
3. The test serves as a foundation upon which may be based some conclusions regarding the heat stability of a lubricant at temperatures at least as high as any ever likely to be encountered in service.
4. The test permits the determination of these properties on comparatively small samples of materials which in many cases are either quite expensive or of very limited availability.

CORROSION AND RUSTING OF STEEL

Before lubricants may be considered for use in instruments, especially in ball bearings, they must pass tests which show that they do not promote corrosion of steel. It is also important in many cases that the lubricants actually inhibit steel corrosion, and hence tests must be made to evaluate their ability to act in such a beneficial way.

A sample of steel coated with a lubricant which does not protect against or which accelerates moisture corrosion, held at room temperature in air of 100 per cent relative humidity, will show signs of corrosion within a few days. In this simple test lubricants which are not corrosive may be left for weeks in contact with steel without showing any evidence of corrosion. In order to determine their relative ability to inhibit corrosion an accelerated test is employed.

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[5] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1559.

A diversity of accelerated tests has been devised and a number of them have been reported in the literature. Humidity cabinets, water sprays, salt solutions, etc., are employed. Most tests of this type employ a steel test specimen in contact with water and the lubricant at an elevated temperature. A well-known and widely used test is the A.S.T.M. Tentative Method of Test for Rust-Preventing Characteristics of Steam-Turbine Oil in the Presence of Water (D 665 – 44 T).[6]

Most of these tests are of utility in evaluating lubricants, but they require expensive equipment, involve complicated procedure, or call for such large amounts of lubricant that they are not practical for appraising small samples. The following procedure was designed to overcome many objections to the existing tests and yet make it possible for anyone to duplicate the conditions. Steel balls, of the type used in ball bearings, are employed as the test specimens; they are uniform in composition, convenient to work with, and need very little treatment to prepare them for use.

Test Procedure:

The test uses 15-ml. wide-mouth screw-cap bottles for containers and 0.5-in. steel balls as the test specimens. The balls are freed of the manufacturer's protective oil or grease coating by washing them in benzene or petroleum naphtha. They are then polished with a levigated alumina on a chamois in order to provide a fresh, clean surface. The alumina is removed by washing the balls in distilled water and swabbing with wet cotton. The washed balls are rinsed in ethyl acoholl and dried in an oven at 105 C.; they are then stored in a desiccator containing magnesium perchlorate until used.

A clean steel ball is placed in a test bottle and covered with 5 ml. of the lubricant. With the cap on, the bottle is suspended in an oil bath at 60 C. (±1 C.). After 1 hr. 5 ml. of distilled water is added, and the mixture is shaken and then allowed to separate into two phases. Usually the oil floats on the water in such a way that it is not in contact with the ball. If the oil is heavier than water and separates readily in such a manner that the ball is not in contact with the water, half of the oil is removed. The bottle is returned immediately to the oil bath.

An examination is made at intervals, and the condition of the ball and the appearance of the layers of oil and water are observed. The test is continued until at least one fourth of the surface of the ball is corroded or until a period of 500 hr. has elapsed. The test is then stopped. The time of testing and the condition of the oil, the water, and the ball are recorded. The ball is washed with a 1:1 mixture of benzene and isopropanol. It is then coated with a dilute collodion solution and filed for reference.

Discussion:

A lubricant with no ability to inhibit corrosion will permit the ball to corrode visibly in several hours. At the end of 24 hr. the ball will be

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[6] 1944 Book of A.S.T.M. Standards, Part III, p. 1283.

TABLE II.—ACCELERATED RUST TEST DATA.

Lubricant	Duration of test, hr.	Condition at End of Test		
		Oil	Water	Ball
White mineral oil (U. S. P. liquid petrolatum, viscosity = 19 cs. at 100 F.)	24	Clear	Brown, rusty sediment	Rusted
White mineral oil + 1.0 per cent stearic acid	360	Clear yellow	Cloudy yellow-brown. Rusty sediment	Rusted
Commercial compounded lubricant containing rust inhibitor.	500	Clear	Clear	Unaffected

badly corroded. The water will contain sediment, usually rust-colored but sometimes brown or black. If the composition of the lubricant is such that it is capable of preventing corrosion, there will be no change in the appearance of the steel ball for 500 hr. or more under the conditions of the test.

Occasionally, as a result of incomplete cleaning of the steel balls or of the containers, a test run on a rust-inhibiting lubricant will fail. For this reason, duplicate check runs are made on all samples which fail; and if neither sample corrodes, the lubricant is considered to have passed the test.

Tests have been run by this procedure on many of the materials commonly used as lubricants. Typical test data are presented in Table II.

EVAPORATION

The rate of evaporation of lubricating oils for instruments was measured by bubbling nitrogen through a sample contained in a specially designed cell. This test was devised (1) to obtain results in less time than testing oil in service requires, (2) to study the evaporative property separate from other characteristics, for example, oxidation, (3) to detect oils whose composition changes appreciably as evaporation proceeds, and (4) to arrange the oils in order of volatility. The test is an improved form of that devised by Barker and Urie (2); but the temperature of evaporation was 70 C. in the test described here.

It was desired that the test, in addition to fulfilling these requirements, yield reproducible, easily

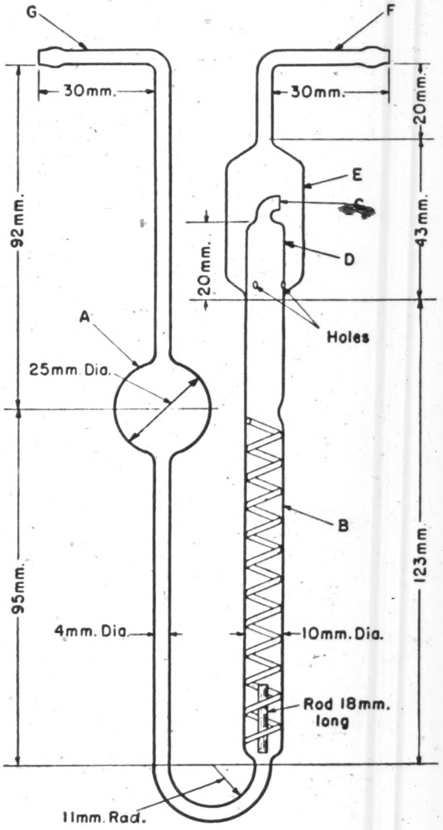


FIG. 3.—Evaporation Test Cell.

understood results and that it be simple but sufficient. Acceleration and a fair degree of precision were attained by causing the weight loss to approach closely the limit set by saturating the surrounding gas with oil vapor. Saturation also enables the findings to be expressed as percentage evaporated, as evaporation rate, or as vapor pressure, the first two of which are especially easy to comprehend. The use of nitrogen eliminates oxidation or other chemical action. A high rate of gas renewal compared to that found in most aircraft instruments shortens the test from months under service conditions to about ten days. A varying rate of evaporation indicates change of composition. Such a change reveals that, although the oil may meet specifications while it is in its original closed container, it begins to differ therefrom immediately on being used.

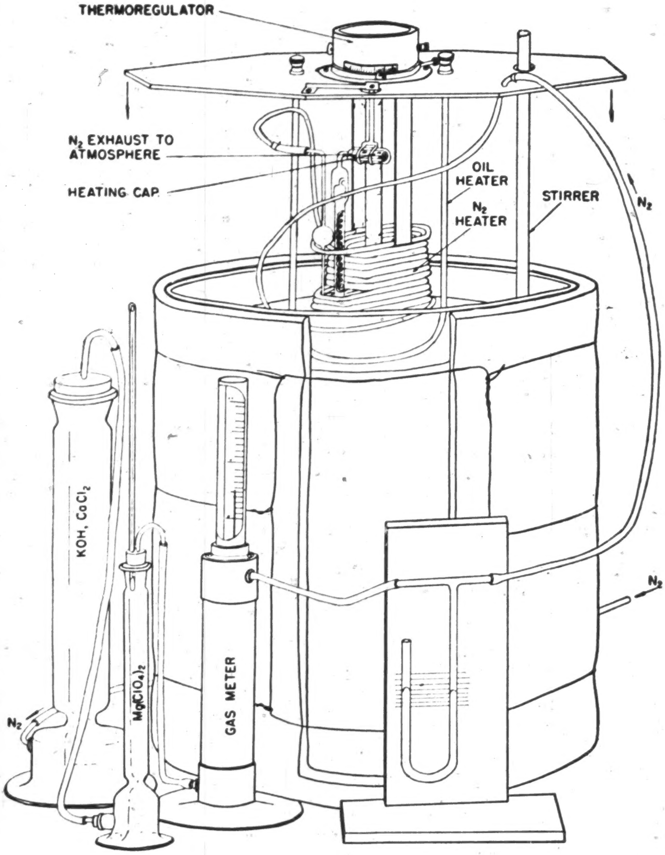

THERMOREGULATOR
N_2 EXHAUST TO ATMOSPHERE
HEATING CAP
OIL HEATER
N_2 HEATER
STIRRER
N_2
$KOH, CaCl_2$
$Mg(ClO_4)_2$
GAS METER
N_2
N_2

FIG. 4.—Evaporation Test Apparatus.

Apparatus:

The cell in which the sample is held for evaporation is shown in Fig. 3. The bulb *A* is a trap to prevent loss of oil if the gas flow should happen to reverse. The spiral in column *B* furnishes surface to saturate the nitrogen with oil vapor; the rod at the lower end is a safeguard against channeling. The inside cage *D* narrows down the column to the hook *C*, which, by increasing the gas velocity and directing the gas against the baffle *E*, generally prevents loss of unvaporized sample as entrainment or foaming through the top arm and exit *F*.

The accessory pieces of apparatus are related as shown in Fig. 4. Condensation in the top arm is prevented by a heating cap. The cap, which consists of a section of copper pipe, dips into the hot oil bath and conducts heat up over the arm, providing a heated region around this otherwise exposed part of the evaporation tube.

Procedure:

1. The evaporation tube is cleaned and weighed.
2. Into the receiving arm *G* is drawn 1 ± 0.2 g. of oil and the filled tube is weighed to 0.2 mg.
3. The tube with the sample is placed into the holder, which also serves as the nitrogen preheater.
4. The nitrogen connection is made (2-in. lengths of neoprene tubing); the top arm cap is put into place; and the apparatus is lowered into the thermostat.
5. The nitrogen flow is adjusted to 0.5 cu. ft. per hr. according to the flowmeter.
6. After a time interval (depending upon the volatility of the oil but usually not less than 18 hr.) the tube is removed, cleaned on the outside, and weighed. To avoid entrance of cleaning solvent into the evaporation tube appropriate precautions must be taken.
7. The tube is replaced and evaporation continued. The procedure is repeated daily until sufficient evaporation is adjudged to have occurred.
8. The results calculated from the data obtained are generally reported graphically.

Derivation of Equations:

It was thought that in instrument work the principle of plotting percentage of original sample remaining as a function of time would be more suitable than reporting vapor pressure only, and therefore results have been expressed accordingly.

The percentage of the sample remaining at any given time is

$$\frac{m_t}{m_0} \times 100 = N \text{..........} (1)$$

This value, N, is plotted as the ordinate.

The abscissa, or time expression, is derived from an adaptation of the equation used in the gas saturation method of determining vapor pressure. As the vapor pressure of the oil is low, the volume of vapor, v, is taken as that of the nitrogen alone (3):

$$M = \frac{(m_0 - m_t)RT}{pv} \text{.......} (2)$$

By correcting the experimental data to a set of "standards," the extent of evaporation may be revised for variations in sample size and gas rate. Let the standard sample size, m_s, be 1.0000 g. and the standard gas rate, q_s, be 25.0 liters per hr. (N.T.P.). Substituting in Eq. 2, which must be true for any sample

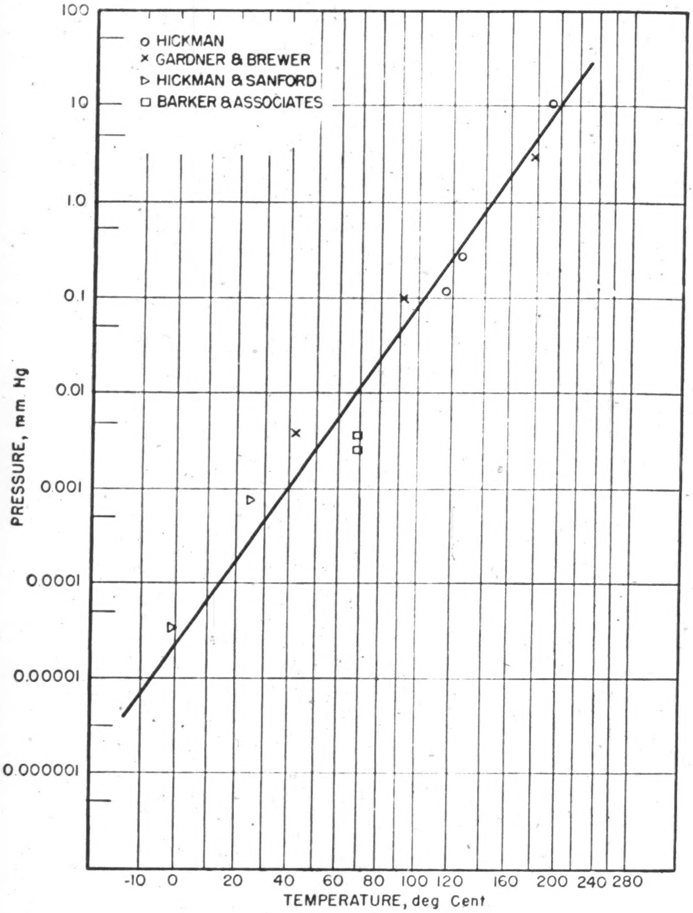


FIG. 5.—Vapor Pressure — Temperature Curve of Di-*n*-Butyl Phthalate.

size and for any gas rate (provided saturation is realized), we find

$$M = \frac{m_s(100 - N)RT}{100pq_st_s \times \frac{T}{273} \times \frac{760}{P}} \quad ..(3)$$

Equation 2 must be true also for the conditions under which the experiment was run. Therefore,

$$M = \frac{m_0(100 - N)RT}{100\,pqt \times \frac{T}{273} \times \frac{760}{P}} \quad ...(4)$$

Then, from Eqs. 3 and 4,

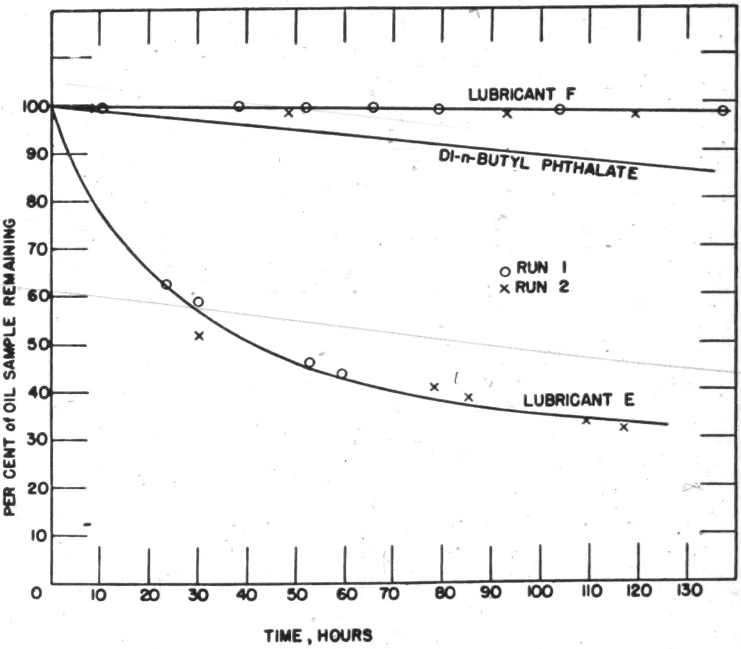
$$\frac{q_st_s}{m_s} = \frac{qt}{m_0}, \text{ or } t_s = \frac{qtm_s}{q_sm_0} \quad ......(5)$$

This value, t_s, is plotted as the abscissa. Under the following set of conditions, t_s represents an actual as well as a calculated value of time:

1. The composition of the oil remains constant during the test.
2. The initial sample is 1.0000 g.

3. The gas rate is 25 liters per hr. (N.T.P.).

If the oil composition remains unchanged during evaporation, Eq. 3 is the equation of the evaporation curve.



Fig, 6.—Evaporarion Rate Curve.

Results:

Comparison of the vapor pressures (0.00262 and 0.00385 mm. Hg for separate runs) found for di-*n*-butyl phthalate with other reported values (Figs. 5 and 6) shows that the apparatus described practically saturates the gas and thus gives results closely related to vapor pressure (see Fig. 5 for the comparison).

Curves *E* and *F* in Fig. 6 represent two runs on each of two different oils. These two curves, plotted from the data in Table III, indicate the reproducibility or degree of precision given by this test. Curve *E* exemplifies the results from an oil whose properties alter rapidly, as revealed by the changing slope—an oil expected to require frequent renewal in the instruments. Curve *F* shows results given by an improved lubricant.

Nomenclature:

m_0 = initial weight of experimental sample used,

m_s = initial weight of "standard" sample,

m_t = weight of experimental sample remaining at time *t*,

M = molecular weight of the oil,

N = percentage of initial sample remaining at time *t*,

p = vapor pressure of the oil,

TABLE III.—EVAPORATION TEST.

Point Number	Initial Sample Weight, m_0, g.	Elapsed Time, t, hr.	Observed Data		Calculated Data	
			Weight of Tube and Sample, g.	Average Gas Rate (N.T.P.), R_a, cu. ft. per hr.	Residue from Standard 1.0000-g. Sample, cg.	Calculated Time, t_s, hr.
			OIL E, RUN 1			
	0.8560		28.7385 (initial)			
1		19.2	28.4197	0.94	62.8	23.9
2		24.1	28.3850	0.94	58.7	30.0
3		42.5	28.2774	0.94	46.2	52.8
4		47.9	28.2581	0.94	43.8	59.5
			OIL E, RUN 2			
	0.7299		28.6124 (initial)			
1		21.0	28.2590	0.94	51.6	30.6
2		41.8	28.1770	0.94	40.4	78.5
3		49.4	28.1582	0.94	37.8	85.0
4		66.2	28.1249	0.94	33.2	109.6
5		71.3	28.1158	0.94	32.0	117.0
			OIL F, RUN 1			
	0.9528		28.9018 (initial)			
1		19.0	28.8960	0.47	99.5	10.9
2		68.8	28.8918	0.47	99.0	39.6
3		94.2	28.8897	0.46	98.8	53.0
4		118.0	28.8878	0.48	98.6	69.2
5		141.8	28.8860	0.48	98.4	83.1
6		186.2	28.8838	0.46	98.2	104.8
7		234.0	28.8814	0.47	98.0	134.5
			OIL F, RUN 2			
	0.8829		28.8286 (initial)			
1		16.0	28.8241	0.47	99.5	9.6
2		87.8	28.8172	0.43	98.8	48.4
3		164.8	28.8103	0.45	97.9	95.1
4		207.5	28.8062	0.47	97.5	125.0
5		257.0	28.8015	0.47	97.0	155.0

P = total pressure under which the evaporation is carried out,
q = actual gas rate used in test; corrected to normal temperature and pressure,
q_s = gas rate used as "standard" for calculations,
R = gas constant,
t = time during which evaporation occurs,
t_s = calculated time of evaporation, and
T = temperature of oil and nitrogen in zone of contact.

SPREADING

In the lubrication of pivot-type bearings, such as are found in clocks and watches and in some aircraft instruments, the tendency of the oil to spread is important. There are many ways to approach the problem of determining the spreading tendency of a lubricant. Other than testing in actual instruments, one of the best indications of nonspreading characteristics is obtained by measuring the contact angle between the lubricant and the solid surfaces to be lubricated. Theoretically, any contact angle greater than zero indicates that the liquid will not spread on the surface in question. Experience has shown that the greater the contact angle the less is the tendency of the liquid to spread. A contact angle of 5 to 10 deg. is probably sufficient to insure a nonspreading lubricant. The next most useful property in predicting the tendency of the lubricant to stay in the bearing is its surface tension. The greater the surface tension the less is the tendency for the oil to leave the bearing. Generally nonspreading oils have a minimum surface tension of 35 dynes per centimeter.

These tests give no evidence of the effect of aging of the oil or of changing the surfaces on the spreading tendency of the lubricant. To fill this gap in the test procedure, an "extended spreading test" has been introduced in which a drop of oil approximately 1 mm. in diameter is placed on a highly polished steel surface and its change in diameter is measured periodically. One set of oil samples is maintained in an anhydrous atmosphere (in a desiccator with magnesium perchlorate) and a duplicate set is kept in a saturated atmosphere (enclosed in a container over water, but not immersed). In the saturated atmosphere condensation occurs on the steel surface and spreading commences in much less time than it does in dry air.

Experimental Procedure:

Contact Angle Measurement.—There are several methods of measuring contact angles of liquids on solid surfaces, but probably the most desirable procedure for the present problem is the measurement of the dimensions of a sessile drop on the surface in question (**2, 4, 5, 6, 7, 8**). The dimensions of the drop are determined by means of a low-power microscope equipped with a

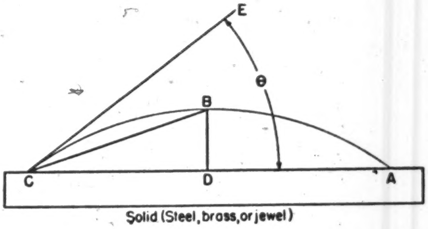


FIG. 7.—Contact Angle of Liquid on Solid.

filar micrometer eyepiece. If the drops are small, their surfaces will be portions of spheres. Then angle θ, the contact angle, will be twice the angle BCD, which is $2 \tan^{-1} (BD/CD)$ (Fig. 7).

The preparation of the solid surfaces is the most important factor involved in reproducing contact angle measurements. The materials used for the test surfaces consist of a piece of $\frac{3}{4}$-in. brass rod, a sample of $\frac{1}{2}$-in. hardened steel drill rod, and a synthetic ruby. The flat end surfaces of the brass and steel samples are polished with metallographic polishing papers Nos. 00, 000, and 0000, in the order named, and finally with Fisher's "Gamal" polishing alumina on a wheel covered with heavy broadcloth. The ruby surface is cleaned by rubbing with rouge on a chamois. After the final polishing, the surfaces are washed with water, swabbed with wet absorbent cotton, and rinsed thoroughly with distilled water. The samples are finally rinsed with acetone before drying for 15 min. in an oven at 105 C. The specimens are allowed to cool to room temperature in a desiccator containing anhydrous magnesium perchlorate.

TABLE IV.—CONTACT ANGLES OF LUBRICANTS.

Lubricant	Surface	Diameter of Drop, D, mm.	2 × Height of Drop, $2h$, mm.	$2h/D$	$\tan^{-1} 2h/D$	θ, deg.	
N-22a	Brass	0.828	0.167	0.202	11	22	23
		0.886	0.189	0.214	12	24	
		0.886	0.193	0.218	12	24	
	Steel	0.911	0.149	0.164	9	18	21
		0.969	0.185	0.193	11	22	
		0.976	0.189	0.194	11	22	
	Ruby	1.050	0.224	0.213	12	24	25
		1.060	0.218	0.206	12	24	
		1.170	0.289	0.247	14	28	
Commercial watch oil "G"	Brass	0.928	0.160	0.173	10	20	22
		0.851	0.144	0.169	10	20	
		0.727	0.164	0.225	13	26	
	Steel	0.836	0.168	0.201	11	22	21
		0.791	0.140	0.177	10	20	
		0.825	0.160	0.194	11	22	
	Ruby	0.747	0.164	0.220	13	26	26
		0.767	0.176	0.229	13	26	
		0.789	0.180	0.228	13	26	
Commercial watch oil "H"	Brass	1.035	0.0967	0.0935	5	10	10
		1.198	0.0934	0.0780	5	10	
		1.221	0.1035	0.0845	5	10	
	Steel	1.261	0.0868	0.0686	4	8	8
		1.302	0.0900	0.0690	4	8	
		0.988	0.0666	0.0678	4	8	
	Ruby	1.000	0.260	0.260	15	30	27
		1.061	0.239	0.225	13	26	
		1.080	0.243	0.225	13	26	

The lubricant is dispensed from a microburette constructed for delivering reasonably uniform drops on the surface being used. With considerable experience, uniform drops of a liquid can be placed on a solid surface with an ordinary steel pen. Generally three drops of the liquid are put on each surface and an average of the contact angles is reported if the difference between any two on the same surface is not greater than 5 deg. If the difference is greater than 5 deg., the surface is cleaned and polished and the measurement is repeated. A drop of liquid 1 mm. across is a satisfactory size.

Extended Spreading Test.—This

test is conducted on steel only but could be made on any solid. For this purpose, ½-in. steel rollers such as are commonly used in roller bearings may be employed as the surface specimens. One plane end of each roller is prepared exactly as the steel surface for contact angle measurements. One sample is placed in a desiccator (Fig. 8) and the other one is put in a closed vessel containing water to maintain the air saturated with water vapor. The diameter of the drop of liquid is measured periodically, usually once a month. This test is conducted only on those lubricants which have passed all other tests and are indicated to be nonspreading from contact-angle measurements.

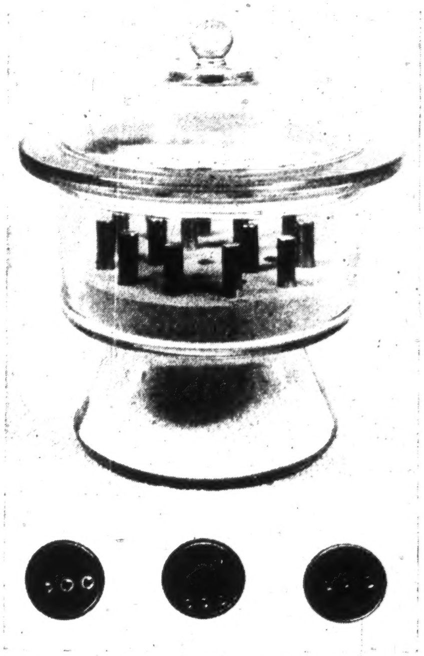


FIG. 8.—Extended Spreading Tests.

Results of Typical Measurements:

Some typical measurements of contact angles are recorded in Table IV. Similarly, measurements as determined in the extended spreading test are presented in Table V.

TABLE V.—EXTENDED SPREADING TEST ON STEEL.

Lubricant	Diameter of Drop, mm.		Time days
	Anhydrous Atmosphere	Saturated Atmosphere	
N-22a.	1.25	1.37	0
	1.55	3.0	123
	1.43	Spread	152
	2.23	. . . . .	395
	Spread	. . . . .	425
Commercial watch oil "G".	2.12	1.30	0
	2.08	1.29	124
	1.94	Spread	212
	2.05	. . . .	755
N-32c.	1.30	1.29	0
	1.44	Spread	150
	Spread	. . . .	180

COEFFICIENT OF FRICTION OF STEEL ON SAPPHIRE, LUBRICATED

Fine mechanisms which contain pivot-type bearings, such as are found in watches, clocks, and some aircraft instruments, are affected greatly in operation by the presence of friction. For this reason, extreme care is used in the design, material selection, and finish of pivot-type instrument bearings.

Because it is difficult to measure quantitatively the friction effect in an instrument, and as it is almost impossible to obtain an accurate representative value without making similar measurements on a large number of instruments, it was decided to design and construct a simple device for measuring the friction of two rubbing surfaces. A number of machines for measuring the effects of lubricants on the coefficient of friction under conditions of boundary lubrication have been described in the literature (**8, 9, 10, 11, 12, 13**).

Design of the Instrument:

After careful consideration of the characteristics of the previously described machines, it was decided to design and construct a pendulum type of instrument to measure the "oiliness" of lubricants. This type of machine was chosen because it makes available an oscillatory motion similar to that found in timepieces, the most numerous of the instruments containing lubricated pivot-type bearings. The design was developed to produce a linear speed between the bearing surfaces in the range encountered in the balance wheels of clocks and watches.

The theory of the measurement of friction by the damping of the oscillation of a pendulum is quite simple if the following assumptions are made:

1. That the normal force, F, on the bearing is not appreciably affected by the centrifugal force.
2. That the normal force, F, is not greatly dependent upon the amplitude.
3. That the damping effect by air resistance is negligible.
4. That the moment of friction, M, of the pendulum about its point of support may be considered constant.

Under these conditions,

$$M = WL\,\alpha_0$$

where:

W = weight of the pendulum,
L = distance of the center of gravity from the point of suspension of the pendulum, and
α_0 = decrease in the amplitude, α, brought about during one-quarter period by friction damping.

Observing full periods, the value of the amplitude, A_n (radians), of the nth period is $A_0 - 4n\,\alpha_0$, where A_0 is the initial value of the amplitude and n is the number of full periods. Then

$$\alpha_0 = \frac{A_0 - A_n}{4n}$$

and

$$M = WL\left(\frac{A_0 - A_n}{4n}\right)$$

The force of friction on each plane surface is $R = f \times F$, where f is the coefficient of friction and F is the normal force on that surface (Fig. 9). Therefore

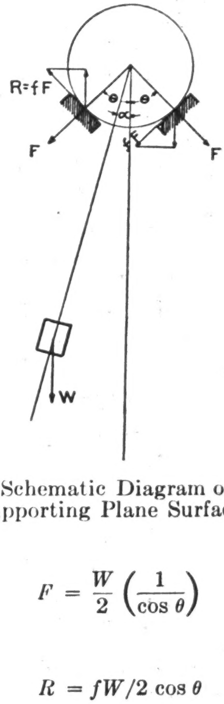


FIG. 9.—Schematic Diagram of Ball and Supporting Plane Surfaces.

$$F = \frac{W}{2}\left(\frac{1}{\cos\theta}\right)$$

and

$$R = fW/2\cos\theta$$

where θ is the angle between the vertical and the radius of the ball at its point of tangency with the plane bearing surface.

Then, since $M = 2(R \cdot r)$, where r is the radius of the ball:

$$M = \frac{frW}{\cos\theta} = LW\left(\frac{A_0 - A_n}{4n}\right)$$

and

$$f = \left(\frac{L \cos \theta}{r}\right) \frac{A_0 - A_n}{4n}$$

The constants for the particular instrument which was constructed are as follows:

$L = 5.18$ in.,
$\theta = 45$, and
$r = 0.250$ in.

Substituting these values in the formula for the coefficient of friction,

$$f = \frac{(5.18)\ (0.7071)}{0.250} \left(\frac{A_0 - A_n}{4n}\right) = 14.64 \left(\frac{A_0 - A_n}{4n}\right)$$

A general view of the instrument is reproduced in Fig. 10. The details of the pendulum and the sapphire bearing are given in Fig. 11.

Experimental Procedure:

It should be emphasized that measurements of oiliness are relative and that such determinations must be conducted under carefully controlled conditions.

The steel ball is prepared by washing with a suitable solvent to remove the previously tested lubricant, and polishing with rouge on moist chamois. The ball is washed with distilled water, swabbed with absorbent cotton, rinsed with alcohol or acetone, and dried for 15 min. at 105 C. before cooling in a desiccator. The plane (sapphire) bearing surfaces are prepared by washing with a suitable solvent to remove the previously tested lubricant and cleaning the surface by buffing with levigated alumina (Fisher's "Gamal") on moist chamois. The plates are washed with distilled water, swabbed with wet cotton, rinsed with alcohol or acetone, and dried for 15 min. at 105 C. before cooling in a desiccator. The surfaces of the cleaned steel ball and sapphire jewels are never allowed to come into contact with the fingers.

The lubricant is applied to the surface by transferring a few drops from the container to the jewels by a glass rod drawn to a point at one end. The drops of the lubricant are wiped over the surface by a piece of absorbent cellulose (such as "Kleenex"). The latter, which is now moist with the lubricant, is also wiped over the bearing surface of the steel ball. The pendulum is then suspended with the ball seated on the two sapphire jewels and measurements are begnu by releasing the pendulum from any

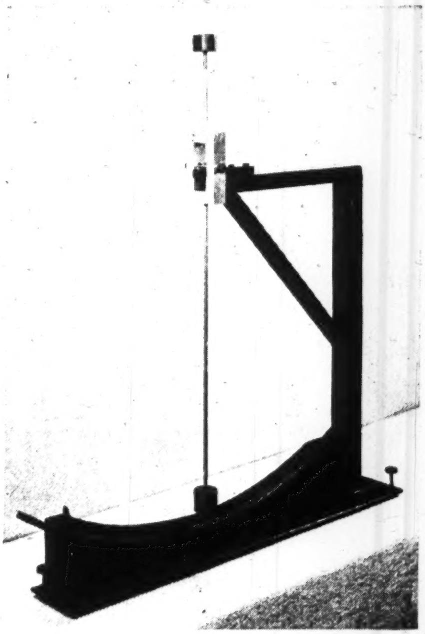


FIG. 10.—Pendulum for Measuring Oiliness.

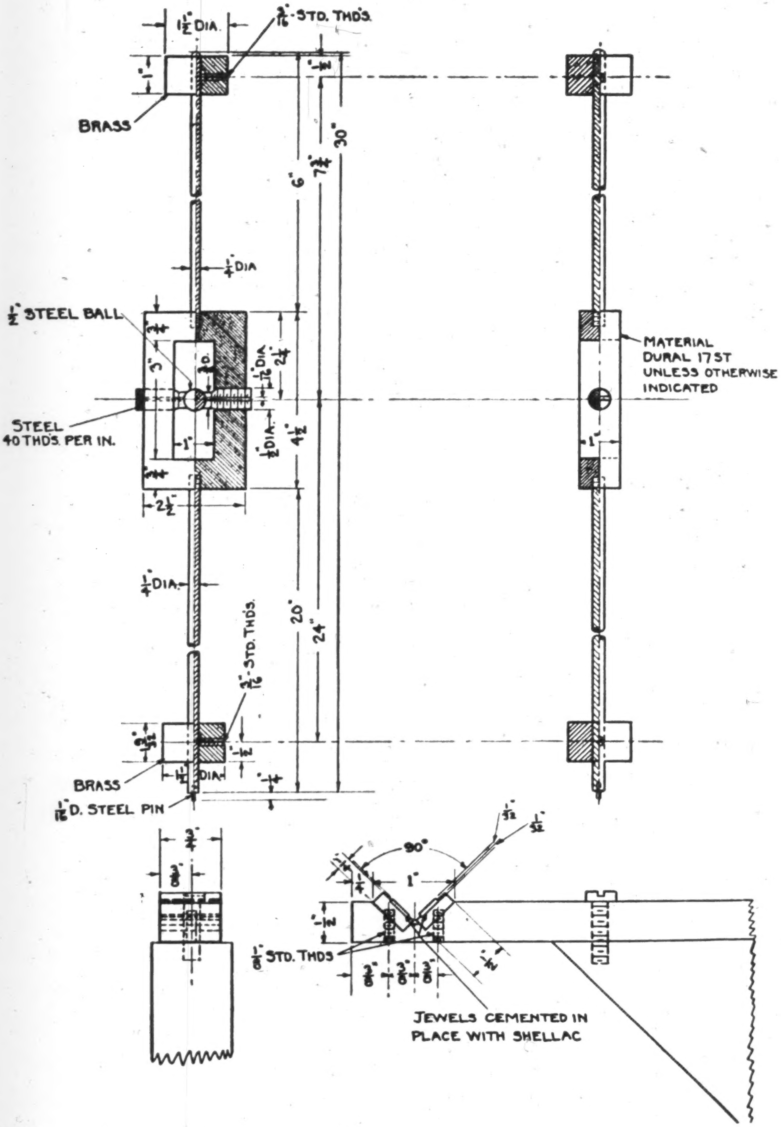

1½" DIA.
5/16" STD. THDS.
BRASS
1"
6"
7½"
30"
¼" DIA.
½" STEEL BALL
3"
2¼"
1/16" DIA.
STEEL
40 THDS. PER IN.
1"
4½"
2½"
MATERIAL
DURAL 17 ST
UNLESS OTHERWISE
INDICATED
¼" DIA.
20"
24"
5/16" STD. THDS.
BRASS
1/16" D. STEEL PIN
½" DIA.
¾"
⅜"
90°
1"
⅛" STD. THDS
JEWELS CEMENTED IN
PLACE WITH SHELLAC

FIG. 11.—Detailed Drawing of Pendulum and Bearing.

desired initial amplitude and observing successive amplitudes. It is most convenient to record the amplitude of every third swing. Tenths of a degree can be estimated on the scale. The average of five determinations is taken as the final value. Table VI contains the values of the coefficients of friction from some typical lubricants.

Acknowledgments:

Many of the testing procedures

TABLE VI.—COEFFICIENTS OF FRICTION OF STEEL ON SAPPHIRE, LUBRICATED.

Lubricant	Swing Number	Amplitude of Swing (Average of five), deg.	Coef. of Friction
Light mineral oil..	0	25	
	3	19.8	0.1108
	6	14.7	0.1100
	9	9.6	0.1097
	12	4.4	0.1100
Castor oil........	0	25	
	3	21.6	0.0725
	6	18.2	0.0725
	9	15.0	0.0712
	12	11.8	0.0705
Porpoise jaw oil...	0	25	
	3	20.4	0.0980
	6	16.0	0.0959
	9	11.6	0.0954
	12	7.2	0.0950

studied and applied in this research were adopted during Mr. Barker's incumbency of the industrial fellowship supported in Mellon Institute by the Elgin National Watch Co.—a comprehensive project on synthetic watch lubricants that preceded and supplied basic guiding lights for the program of the Instruments Branch of the Bureau of Aeronautics. The authors are highly appreciative of the generous cooperation of the scientific staff of the Elgin National Watch Co. and of personnel of the Instruments Branch, Bureau of Aeronautics. The Aeronautic Instrument Section of the National Bureau of Standards has also been of material aid during the progress of the investigation.

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GREASE ANALYSIS—AN ANALYSIS OF THE A.S.T.M. METHOD AND SUGGESTIONS FOR IMPROVEMENT*

By Robert S. Barnett,[1] Walter Scott Palmer,[1] and Harry Levin[1]

This paper deals with the analysis of lubricating greases, including separation and determination of the main components, such as fatty acids from soap, mineral oil, etc. The A.S.T.M. Standard Methods of Analysis of Grease (D 128-40)[2] are those generally accepted for these determinations. Their wide recognition is apparent from their incorporation into the following publications: Federal Standard Stock Catalog, VV-L-791b, February 19, 1942, p. 178; Institute of Petroleum, Standard Methods, Sixth Edition, 1945, p. 23; and "Lubricating Greases: Their Manufacture and Use," E. N. Klemgard, p. 799 (1937). They have also been approved as American Standard by the American Standards Association (ASA No. Z11.16-1940).

A.S.T.M. Methods D 128 were issued in 1927 and remain largely unaltered. Since their adoption, grease research and technology have expanded greatly, the "art" concept has almost disappeared from the picture, there is less tendency to keep formulas secret, and methods of analyses are more closely examined.

The need for more comprehensive and reliable methods of grease analysis results from these developments. Such a method should serve at least the following types of samples:

1. Experimental and production samples including such components as lithium soap and co-existing free acid and free alkali.

2. Used or deliberately degraded greases; to determine the extent of deterioration as, for example, after accelerated laboratory tests.

3. Samples for referee analyses to determine composition. This phase is less important than previously, since the trend is to performance tests for determining grease quality.

This paper presents modifications of the A.S.T.M. Methods D 128 which have been used successfully in our laboratories for the analyses of thousands of greases from research and production. The modifications render the basic principles of the Methods D 128 more comprehensive, its manipulations more practical, and improve precision; in addition, methods are given for determining co-existing free acid and free alkali, lithium in greases, qualitative presence of soap, all of which amplify and augment the utility of the A.S.T.M. method. Mention is also made of rapid simple procedures for estimating soap content, etc. when much is known about the nature of the sample, as in manufacturing control.

Comparison of the A.S.T.M. and the Proposed Methods

A.S.T.M. Methods D 128 – 40 involve decomposing the grease with hy-

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[1] Assistant to Superivsor, Products Application Service, The Texas Company, Beacon, N. Y.; Chemist in Charge Analytical Laboratory, The Texas Company, Port Arthur, Tex.; and Supervisor, Products Analysis, The Texas Company, Beacon, N. Y., respectively.

[2] 1944 Book of A.S.T.M. Standards, Part III, p. 182.

drochloric acid and separating the total oily organic acids by alkali extraction from the nonacidic oil components using immiscible solvents in a counter-extraction operation. The modified method, given for convenience in detail in Appendix I, follows the same general procedure and form, but incorporates changes which the authors have found improve the precision and ease of analytical operations.

A summary of the principal modifications follows:

Fillers (*Section 10*):[3]

The A.S.T.M. method involves digestion of the sample with hydrochloric acid, filtration, and washing with naphtha and alcohol. This does not determine acid-soluble fillers, such as chalk, mineral constituents of graphite, etc.

The modified method utilizes inert solvents which dissolve the grease and leave fillers as residue.

Soap (*Section 12*):

The A.S.T.M. method specifies that the primary decomposition of the grease with dilute hydrochloric acid be conducted in a separatory funnel, presumably at atmospheric temperature. It is much more expedient to decompose it hot and in the presence of benzene in an Erlenmeyer flask under a reflux condenser. Subsequently, the benzene is evaporated, and the mixture cooled and transferred to a separatory funnel with naphtha, whenceforth the usual separations are effected.

The A.S.T.M. method specifies that in calculating soap from the pertinent fraction of gravimetrically-determined fatty acids, the ash analysis should be used as the guide if more than one base is present. We supplement this with an actual qualitative test for soap because serious error could result if, for example, a sodium soap grease had a chalk filler, in which case two bases will be found in the analysis of the ash, though the soap of only one base is present.

To keep pace with the growing use of lithium as soap base, a method is included for its determination in lithium-sodium soap greases (Appendix II).

Glycerin (*Section 13*):

The A.S.T.M. method provides only a qualitative separation of glycerin. It is found that a quantitative determination is frequently useful, so a method involving periodate oxidation has been included. It is subject to less interference than dichromate procedures.

Petroleum Oil (*Section 15*):

Small amounts of soap resulting from the extractive separations of the A.S.T.M. method commonly remain dissolved or entrained in the naphtha solution containing the petroleum oil fraction. If not eliminated, this soap may affect the viscosity and other constants of the separated mineral oil. In the modified method, the naphtha solution is acidified with hydrochloric acid and then washed free of mineral acid with water. The petroleum oil separated in this fashion contains a minute amount of fatty acid, which influences its properties much less than the equivalent amount of soap. The quantity of petroleum oil is corrected for the small amount of fatty acid on the basis of the neutralization numbers of the petroleum oil fraction and the fatty acids separated in the main soap determination.

Procedure for Dark Greases by Method II (*Section 16*):

Where the grease contains appreciable asphaltic or tarry matter, the A.S.T.M. method requires decomposition with granulated $KHSO_4$ instead of hydrochloric acid. It is practically impossible to accomplish this with many greases, particularly those containing little or no water. It is like attempting to make intimate contact between lumps of dry solids. We find that digestion with $KHSO_4$ in the presence of small additions of water and benzene completely decomposes the grease, if it is desired to follow the A.S.T.M. method as closely as possible. Subsequent evaporation of these solvents leaves a residue which can be extracted in Soxhlet type apparatus required in the A.S.T.M. method.

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[3] The sections within parentheses refer to A.S.T.M. Standard Methods D 128 - 40, see footnote 2.

The method presented in this paper, by decomposing asphaltic and tarry greases with hydrochloric acid in the usual fashion, obviates the need for this complication. Small additions and rinsings of warm benzene are used to carry along any asphaltic or tarry matter which drops out of the naphtha layer during analysis. If it is acidic, addition of alcoholic potash saponifies such tarry acid and it is included in the "fatty acids from soap plus free fatty acid" where, being acid, it properly belongs. Otherwise, it remains in the "unsaponifiable plus neutral saponifiable" component and is finally estimated, either by saponification or by weighing, as unsaponifiable together with the mineral oil, or as neutral saponifiable together with the neutral fat.

The A.S.T.M. method determines asphaltic and tarry matter as a unit; the proposed method provides means, where necessary, for resolving it into three fractions: acidic, saponifiable nonacidic, and unsaponifiable. Such resolution can be especially important; for example, in greases made with stearine pitch.

Free Alkali and Free Acid (Sections 18, 19, and 20); New Method for Co-existing Free Alkali and Free Acid:

During the past ten years it has become evident that free fatty acid and free alkali can exist simultaneously in a lubricating grease. This has been found where free alkali metal carbonates or alkaline earth metal hydroxides are present. Free acid has not been found in the presence of free alkali metal hydroxide, except for lithium.

The determination of free fatty acid and free alkali in greases has been examined in detail by a subcommittee of the National Lubricating Grease Institute. Its report[4] presented a method for the determination of coexisting free acid and free alkali in lubricating greases based on work of Levin, Barnett, and McDowell.[5] It is applicable to alkali and alkaline earth metal soap greases but, like the A.S.T.M. method, is inapplicable to those of weak bases such as aluminum, lead, etc. Basic soaps of alkaline earth metals in grease properly appear alkaline by the proposed method. Preliminary cooperative work by American and Canadian laboratories, both of A.S.T.M. and N.L.G.I. affiliation, confirms the authors' statement on the general superiority of the method.

It is interesting to note that the Institute of Petroleum's "Standard Methods for Testing Petroleum and Its Products" (Sixth Edition, 1945) contains a revised method (I.P.—37/44) which recognizes the probability of a grease containing both free alkali and free acid. This method is quite similar to that already discussed.

The former A.S.T.M. Method (D 128 – 27)[6] utilized a solvent mixture of naphtha and 50 per cent aqueous alcohol in the free acid or free alkali determination. At maximum effectiveness this could determine only the excess of one constituent over the other. In the 1937 revision of Method D 128,[7] 95 per cent alcohol was substituted for 50 per cent alcohol in order to secure better disintegration of water-insoluble greases. This change, however, made impossible the detection of free alkali in many sodium-base greases, as sodium carbonate is insoluble in 95 per cent alcohol in the presence of soap concurrently dissolved, hence shows no alkaline reaction to phenolphthalein. Table I compares the former and present A.S.T.M. methods.

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[4] Gus Kaufman, "Test Methods for Determining Free Acid and Free Alkali in Greases," Subcommittee on Free Acid and Free Alkali, N.L.G.I. *The Institute Spokesman*, January, 1944.

[5] Submitted to Subcommittee IV on Grease of A.S.T.M. Committee D-2 on Petroleum Products and Lubricants, in 1937.

[6] 1927 Book of A.S.T.M. Standards, Part II, p. 355.

TABLE I.—FREE ACID OR ALKALI COMPARISON OF THE FORMER AND PRESENT A.S.T.M. METHODS.

Requirement	Former A.S.T.M. D 128 – 27 (E)	Present A.S.T.M. D 128 – 40 (18, 19, 20) (Free Acid Determination Revised in 1937)
Size of sample, g.	10 to 30	10 to 30
Solvent mixture	75 ml. petroleum ether 50 ml. 50 per cent alcohol	75 ml. petroleum ether 50 ml. 95 per cent alcohol
Indicator	Phenolphthalein	Phenolphthalein
Disintegration	In cold	In cold
Determination of free acidity	If the alcoholic layer is not pink, titrate acidity in cold with 0.5 *N* alcoholic KOH.	
Determination of free alkali	If the alcoholic layer is pink, add 10 ml. of 0.5 *N* HCL reflux for 10 min., back titrate with 0.5 *N* alcoholic KOH.	
Limitations	At maximum effectiveness determines only the excess of one constituent over the other	Misses free alkali entirely in most sodium-base greases, as Na_2CO_3 is insoluble in 95 per cent alcohol. At maximum effectiveness, determines only the excess of one constituent over the other.

Table II illustrates the varied results obtained by these two methods.

In the two tests on ball and roller bearing greases, the higher results for free alkali obtained by Method D 128 – 27 are probably due to premature end point caused by hydrolysis of sodium soap in the dilute alcohol of the method.

TABLE II.—RESULTS OBTAINED BY TWO A.S.T.M. METHODS.

Sample	Product	Type of Soap	A.S.T.M. Method D 128 – 27, per cent	A.S.T.M. Method D 128 – 40, per cent
No. 1	High heat grease	Sodium	0.20 as NaOH	0.28 as oleic acid
No. 2	High heat grease	Sodium	0.20 as NaOH	0.28 as oleic acid
No. 3	Sponge grease	Sodium	0.03 as NaOH	0.14 as oleic acid
No. 4	Experimental grease	Sodium	0.05 as NaOH	0.05 as oleic acid
No. 5	Cup grease	Calcium	0.11 as $Ca(OH)_2$	0.11 as $Ca(OH)_2$
No. 6	Ball and roller bearing grease	Sodium, calcium	0.11 as NaOH	0.04 as NaOH
No. 7	Ball and roller bearing grease	Sodium, calcium	0.19 as NaOH	0.10 as NaOH

TABLE III.—FREE ALKALI AND FREE ACID ANALYSIS OF KNOWNS.

Sample	A.S.T.M. D 128 - 40		Authors' Method for Co-existing Components	
	Operator 1	Operator 2	Operator 1	Operator 2
No. 1.—Light colored substantially neutral sodium soap grease containing added oleic acid (0.1 per cent) and Na_2CO_3 (1.0 per cent)				
Free fatty acid as oleic per cent	None	None	0.1–	0.1–
Free alkali as Na_2CO_3 per cent	None	None	0.9	0.9
No. 2.—Dark green substantially neutral calcium soap grease containing added oleic acid (1.0 per cent) and $CaCO_3$ (0.5 per cent)				
Free fatty acid as oleic per cent	0.5	0.5	0.8	0.8
Free alkali as $CaCO_3$ per cent	None	None	0.5	0.5

It may be expected that carbonate alkalinity would be missed in the newer (D 128 – 40) methods, unless hydroxide is simultaneously present; the latter being sufficiently soluble in 95 per cent alcohol to give an alkaline reaction, would cause one to proceed to the free alkali determination (D 128 – 40).

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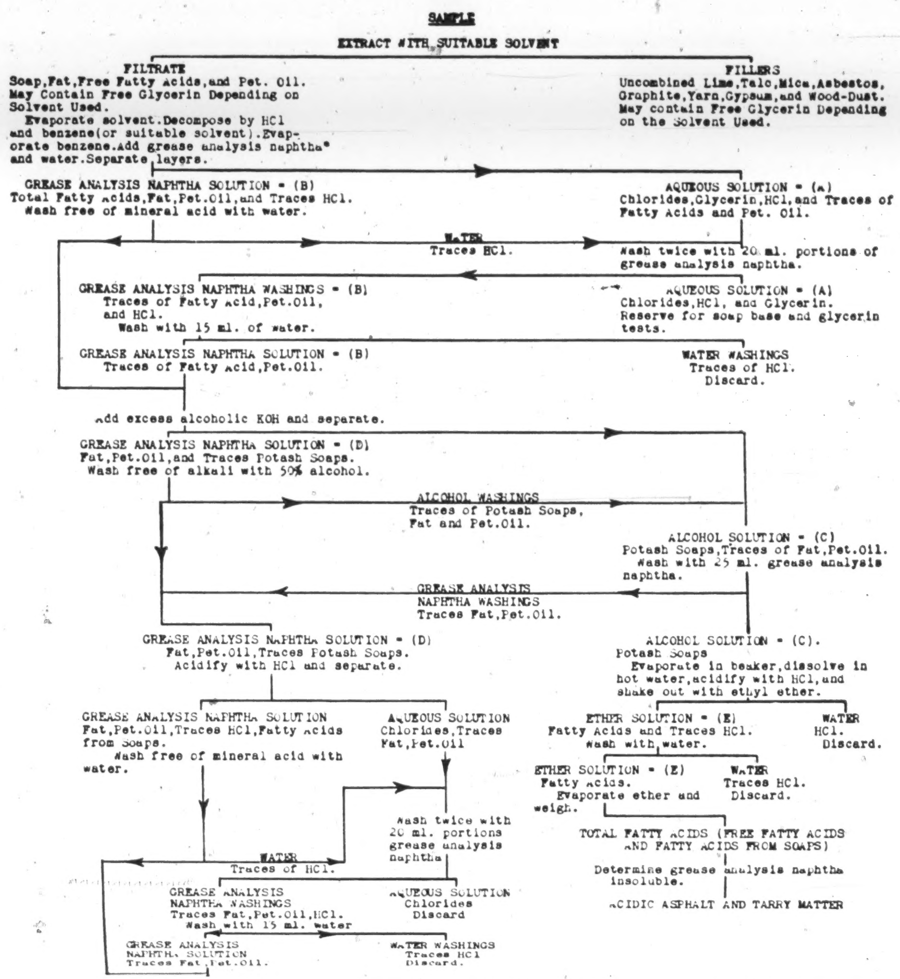
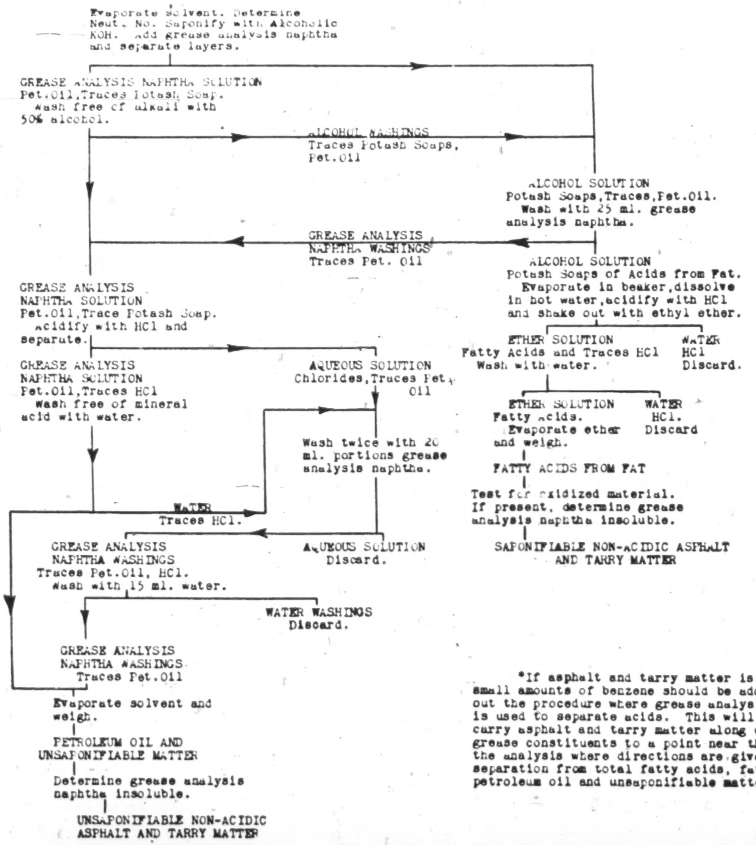
[7] 1939 Book of A.S.T.M. Standards, Part III, p. 159.


SAMPLE
EXTRACT WITH SUITABLE SOLVENT
FILTRATE
Soap,Fat,Free Fatty Acids,and Pet. Oil. May Contain Free Glycerin Depending on Solvent Used.
Evaporate solvent.Decompose by HCl and benzene(or suitable solvent).Evaporate benzene.Add grease analysis naphtha* and water.Separate layers.
FILLERS
Uncombined Lime,Talc,Mica,Asbestos, Graphite,Yarn,Gypsum,and Wood-Dust. May contain Free Glycerin Depending on the Solvent Used.
GREASE ANALYSIS NAPHTHA SOLUTION = (B)
Total Fatty Acids,Fat,Pet.Oil,and Traces HCl.
Wash free of mineral acid with water.
AQUEOUS SOLUTION = (A)
Chlorides,Glycerin,HCl,and Traces of Fatty Acids and Pet. Oil.
WATER
Traces HCl.
Wash twice with 20 ml. portions of grease analysis naphtha.
GREASE ANALYSIS NAPHTHA WASHINGS = (B)
Traces of Fatty Acid,Pet.Oil, and HCl.
Wash with 15 ml. of water.
AQUEOUS SOLUTION = (A)
Chlorides,HCl, and Glycerin.
Reserve for soap base and glycerin tests.
GREASE ANALYSIS NAPHTHA SOLUTION = (B)
Traces of Fatty Acid,Pet.Oil.
WATER WASHINGS
Traces of HCl.
Discard.
Add excess alcoholic KOH and separate.
GREASE ANALYSIS NAPHTHA SOLUTION = (D)
Fat,Pet.Oil,and Traces Potash Soaps.
Wash free of alkali with 50% alcohol.
ALCOHOL WASHINGS
Traces of Potash Soaps, Fat and Pet.Oil.
ALCOHOL SOLUTION = (C)
Potash Soaps,Traces of Fat,Pet.Oil.
Wash with 25 ml. grease analysis naphtha.
GREASE ANALYSIS NAPHTHA WASHINGS
Traces Fat,Pet.Oil.
GREASE ANALYSIS NAPHTHA SOLUTION = (D)
Fat,Pet.Oil,Traces Potash Soaps.
Acidify with HCl and separate.
ALCOHOL SOLUTION = (C).
Potash Soaps
Evaporate in beaker,dissolve in hot water,acidify with HCl,and shake out with ethyl ether.
GREASE ANALYSIS NAPHTHA SOLUTION
Fat,Pet.Oil,Traces HCl,Fatty Acids from Soaps.
Wash free of mineral acid with water.
AQUEOUS SOLUTION
Chlorides,Traces Fat,Pet.Oil
ETHER SOLUTION = (E)
Fatty Acids and Traces HCl.
Wash with water.
WATER
HCl.
Discard.
ETHER SOLUTION = (E)
Fatty Acids.
Evaporate ether and weigh.
WATER
Traces HCl.
Discard.
Wash twice with 20 ml. portions grease analysis naphtha
WATER
Traces of HCl.
TOTAL FATTY ACIDS (FREE FATTY ACIDS AND FATTY ACIDS FROM SOAPS)
Determine grease analysis naphtha insoluble.
ACIDIC ASPHALT AND TARRY MATTER
GREASE ANALYSIS NAPHTHA WASHINGS
Traces Fat,Pet.Oil,HCl.
Wash with 15 ml. water
AQUEOUS SOLUTION
Chlorides
Discard
GREASE ANALYSIS NAPHTHA SOLUTION
Traces Fat,Pet.Oil.
WATER WASHINGS
Traces HCl
Discard.


Evaporate solvent. Determine Neut. No. Saponify with Alcoholic KOH. Add grease analysis naphtha and separate layers.
GREASE ANALYSIS NAPHTHA SOLUTION Pet.Oil,Traces Potash Soap. Wash free of alkali with 50% alcohol.
ALCOHOL WASHINGS Traces Potash Soaps, Pet.Oil
ALCOHOL SOLUTION Potash Soaps,Traces,Pet.Oil. Wash with 25 ml. grease analysis naphtha.
GREASE ANALYSIS NAPHTHA WASHINGS Traces Pet. Oil
ALCOHOL SOLUTION Potash Soaps of Acids from Fat. Evaporate in beaker,dissolve in hot water,acidify with HCl and shake out with ethyl ether.
ETHER SOLUTION Fatty Acids and Traces HCl Wash with water.
WATER HCl Discard.
ETHER SOLUTION Fatty Acids. Evaporate ether and weigh.
WATER HCl. Discard
FATTY ACIDS FROM FAT
Test for oxidized material. If present, determine grease analysis naphtha insoluble.
SAPONIFIABLE NON-ACIDIC ASPHALT AND TARRY MATTER
GREASE ANALYSIS NAPHTHA SOLUTION Pet.Oil,Trace Potash Soap. Acidify with HCl and separate.
GREASE ANALYSIS NAPHTHA SOLUTION Pet.Oil,Traces HCl Wash free of mineral acid with water.
AQUEOUS SOLUTION Chlorides,Traces Pet. Oil
Wash twice with 20 ml. portions grease analysis naphtha.
WATER Traces HCl.
AQUEOUS SOLUTION Discard.
GREASE ANALYSIS NAPHTHA WASHINGS Traces Pet.Oil, HCl. Wash with 15 ml. water.
WATER WASHINGS Discard.
GREASE ANALYSIS NAPHTHA WASHINGS Traces Pet.Oil
Evaporate solvent and weigh.
PETROLEUM OIL AND UNSAPONIFIABLE MATTER
Determine grease analysis naphtha insoluble.
UNSAPONIFIABLE NON-ACIDIC ASPHALT AND TARRY MATTER

*If asphalt and tarry matter is present, small amounts of benzene should be added throughout the procedure where grease analysis naphtha is used to separate acids. This will serve to carry asphalt and tarry matter along with other grease constituents to a point near the end of the analysis where directions are given for its separation from total fatty acids, fat, and petroleum oil and unsaponifiable matter.

FIG. 1.—Scheme of Grease Analysis, Major Separations.

Table III presents data on known samples and it is evident that Methods D 128–40 failed completely to detect sodium carbonate in a sodium soap grease, missed calcium carbonate in a calcium soap grease, and gave low results for free acidity. The authors' method found both coexistant free alkali and free acid with fair accuracy.

Similarly, free $Ca(OH)_2$ in calcium soap grease and free LiOH in lithium soap grease were determinable in the presence of free fatty acids by the authors' method, but not by the A.S.T.M. methods.

CALCULATING AND REPORTING

The modified method includes a section on calculating and reporting of data, including calculation of soap in single-base and mixed-base greases; and a convention for calculation of soap in greases containing soaps of weak bases such as lead, aluminum, etc.

GRAPHIC SCHEME OF GREASE ANALYSIS

The graphic scheme of Methods D 128-40 (Fig. 1)[8] illustrating the major separations has been modified by eliminating Method II (the $KHSO_4$ decomposition procedure) and incorporating the other modifications described in this paper. The revised graphic scheme is given as the accompanying Fig. 1.

Acknowledgments:

The authors value highly the contributions of J. Lyons for adapting the method for lithium; S. J. Morana for adapting the method for glycerin; E. Stein, G. P. Berthelot, and D. T. Burhans for aiding greatly in the establishment of the modified method by careful conduct of numerous analyses; and J. H. Carnett and C. J. Anderson for their contribution to the formal preparation of the method of analysis.

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[8] 1944 Book of A.S.T.M Standards, Part III, p 184

APPENDIX I

METHOD FOR ANALYSIS OF LUBRICATING GREASE

Scope

1. The method is applicable to lubricating greases and comprises procedures for determining the constituents of greases likely to be covered by specifications. The method is also suitable for analyzing used or experimentally-degraded greases. Some modern synthetic ester greases, or greases containing sulfonates, will require modification of the general procedure.

Outline of Method

2. Procedures are described for making the following determinations:

(*a*) *Ash.*—Two methods are given for ash. In one, the sample is burned to constant weight; in the other, the grease is burned until the ash is nearly free of carbon and the residue then sulfated and weighed.

(*b*) *Water.*—Water is determined by a modification of A.S.T.M. Standard Method of Test for Water in Petroleum Products and Other Bituminous Materials (D 95 - 40).[1] This method is used because the apparatus is that employed for petroleum products in general.

(*c*) *Fillers.*—Fillers are determined as the material insoluble in benzene or other suitable inert solvent. Uncombined lime, chalk, graphite, mica, talc, asbestos, gypsum, wood pulp, etc., will be included.

(*d*) *Soap Bases.*—General methods for the detection of soap bases are given. They may be quantitatively determined by accepted procedures. A method is described for determining lithium.

(*e*) *Total Fatty Acids.*—After decomposition of the grease with HCl (10 per cent), free fatty acids and fatty acids from soap are separated by conventional grease solvent methods, asphaltic and tarry matter being carried along in the analysis.

(*f*) *Asphaltic and Tarry Matter.*—If asphaltic and tarry matter is present, it is carried along and separated, depending on its nature, with total fatty acids, fat, or petroleum oil during the course of the separations of these grease constituents. This material may be separated from any or all three of these fractions, as naphtha insolubles. If present in more than one fraction, the asphaltic and tarry matter is taken as the sum of the naphtha insolubles from each fraction. If inert fillers are present, they of couse will appear as naphtha insolubles and are taken into account by separating the asphaltic and tarry material as the benzene soluble.

(*g*) *Petroleum and Unsaponifiable Oil.*—Petroleum and unsaponifiable oil remain after the removal of neutral fat. If asphaltic and tarry matter is present, it is removed as naphtha insolubles, the soluble portion being the petroleum and unsaponifiable oil.

(*h*) *Glycerin.*—Qualitative and quantitative procedures are given for glycerin, oxidation to acrolein for the former, a periodate method for the latter.

(*i*) *Acidity and Alkalinity.*—Methods are given for determining coexisting free alkali and free acid.

(*j*) *Soap.*—Methods are given for determining and calculating soap from the quantities of acids and metals found in the grease.

(*k*) *Miscellaneous.*—The utility of simpler and faster methods is recognized for samples on which a great deal of information is available beforehand, as in manufacturing control.

Reagents

3. (*a*) *Sulfuric Acid* conforming to the current specifications of the American Chemical Society.

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[1] 1944 Book of A.S.T.M. Standards, Part III, p. 293.

(*b*) *Hydrochloric Acid* conforming to the current specifications of the American Chemical Society.

(*c*) *Hydrochloric Acid* (*10 per cent*).—Prepare an HCl solution containing 10 per cent by weight of absolute HCl.

(*d*) *Potassium Hydroxide* conforming to the current specifications of the American Chemical Society.

(*e*) *Methyl Orange Solution.*—Dissolve 0.5 g. of methyl orange in distilled water and dilute to 1 liter.

(*f*) *Phenolphthalein Solution.*—Dissolve 1.0 g. of phenolphthalein in 50 ml. of alcohol, adding 50 ml. of water, and neutralizing before use with KOH solution.

(*g*) *Ethyl Ether* conforming to the current specifications of the American Chemical Society.

(*h*) *Acetone* conforming to the current specifications of the American Chemical Society.

(*i*) *Alcohol.*—Specially denatured alcohol, Formula 30, or its equivalent, neutralized to phenolphthalein with KOH solution.

(*j*) *Grease Analysis Naphtha* having an end point not higher than 200 F. (93 C.). A 125-ml. portion of the naphtha shall not consume more than 0.2 ml. of 0.5 *N* KOH when a blank test is made as follows: Boil 125 ml. of grease analysis naphtha with 10 ml. of 0.5 *N* alcoholic KOH and 50 ml. of neutral 50 per cent alcohol for 1½ hr. on a hot plate, using a glass tube about 7 mm. in internal diameter and 750 mm. in length as a reflux condenser. After cooling, titrate the solution with 0.5 *N* HCl and phenolphthalein. The naphtha shall leave a residue of less than 3 mg. per 200 ml. on evaporation at 212 F.

(*k*) *Benzene* conforming to the current specifications of the American Chemical Society.

Sample

4. (*a*) Stir or mix the original sample until it is uniform.

(*b*) The size of the sample for the determination of fillers, petroleum oil and unsaponifiable matter, soap, and fat shall be 5 to 30 g., depending on the consistency of the grease, which is determined chiefly by its soap content. A 10 to 20-g. sample is usually a convenient amount for No. 3 cup grease, while for semi-liquid greases a larger sample is required. A 5-g. sample is more convenient for dehydrated driving journal greases. If the sample contains a large amount of filler or is difficult to filter, it is advisable to use a small sample (2 to 3 g.) for the determination of filler only and use a larger sample for the remainder of the analysis.

(*c*) Samples need not be weighed more closely than 0.01 g.

ASH

NOTE.—The ash determination should not in general be regarded as of great importance. It does, however, provide a starting material for the qualitative examination for inorganic constituents. (The absence of ash in a soap-type grease indicates an organic base for the soap.) It is often unsatisfactory because of interaction between sodium carbonate derived from the soap with fillers or other grease constituents, as well as likelihood of reaction with the porcelain crucible itself. If much sodium or potassium carbonate is present, the ash is fusible and often encloses carbon, making its complete removal difficult. Results will be low in the presence of easily reducible oxides of volatile metals. There is also uncertainty as to when calcium carbonate has been completely ignited to calcium oxide. Ash determinations made on the same sample in different laboratories are likely to vary widely.

Procedure

5. (*a*) *Rapid Routine Method.*—Place a 2- to 5-g. sample of the grease in a weighed porcelain crucible and weigh to the nearest 0.1 g. If soaps of lead, zinc, and other reducible metals are known to be absent, a platinum crucible is more convenient. Burn off the combustible matter slowly, then ignite the residue until the ash is free from carbonaceous matter. Cool the crucible and contents in a desiccator and weigh.

(*b*) *Alternative Method* (*Sulfated Residue*).—This method gives more concordant results than the routine method outlined in Paragraph (*a*), but requires more time and manipulation. Burn the sample as described in Paragraph (*a*) until the ash is nearly free of carbon. Continue the determination by the procedure in A.S.T.M. Tentative Method of Test for Sulfated

Residue from New Lubricating Oils (D 874 - 46 T).[2].

(c) *Qualitative Examination.*—Dissolve the ash in dilute HNO_3 or HCl and examine for the presence of soap bases by suitable chemical tests, using any conventional scheme of qualitative analysis. Spectrographic methods are particularly helpful to insure that the presence of the less commonly used materials is not overlooked. Useful information may be obtained by closely observing for certain physical characteristics of the ash. Greases containing sodium, potassium, or lithium leave easily fusible ash dissolving completely in water to give a strongly alkaline solution. Calcium or magnesium greases leave white infusible ash of low solubility in water but alkaline in it. Zinc grease yields an ash which is yellow when hot and white when cold. Lead grease leaves an ash which is yellow when cold and commonly contains globules of metal.

(d) *Quantitative Examination.*—Make quantitative determinations by conventional methods for the metals that were found in the qualitative examination of the ash. If the soap of only one metal is present, a quantitative determination is generally unnecessary.

WATER

6. Determine water by A.S.T.M. Method D 95 - 40,[1] modified in accordance with the following Paragraphs (*a*) to (*c*).

(*a*) *Apparatus.*—Use a round-bottom, short, ring-neck, 1-liter flask. The bottom end of the reflux condenser shall be ground off at an angle of 60 deg.

(*b*) *Sampling.*—It is important that the sample be as representative as possible. Do not use outside surfaces which have been exposed to air. When testing hard grease, cut the sample into ¼-in. cubes before weighing.

(*c*) *Procedure.*—The sample weighed into the flask shall be as large as consistent with convenient operation of the test, 100 g. generally being enough. The tendency of some types of grease to swell and puff may govern the maximum weight to be used. Add 500 ml. of solvent to the flask and connect to the trap and condenser. (If a 50-g. sample is used, 250 ml. of solvent in a 500-ml. flask is convenient.) To assure disintegration of the grease, apply heat to the flask in such a manner that for 15 to 30 min. there is only a simmering with just an occasional drop of condensed liquid falling back from the condenser, and then increase heat so that distillate falls from the condenser at a rate of 2 to 5 drops per second. Continue heating until the volume of aqueous liquid in the trap is constant. For greater accuracy, the aqueous distillate may be corrected for glycerin carry-over on the basis of its refractive index.

FILLERS

Procedure

7. (*a*) Transfer a suitable quantity of sample [Section 4 (*b*)] to a 250-ml. Erlenmeyer flask and add sufficient solvent to dissolve the grease and separate the filler. Greases containing more than very low concentrations of water should be dehydrated in an oven before adding the solvent. Hot benzene or xylene alone or in conjunction with methyl or isopropyl alcohol is generally a satisfactory solvent. The aromatic hydrocarbons, methyl alcohol, and isopropyl alcohol frequently are employed successively. Special cases may require the use of other inert solvents, such as esters, halogenated hydrocarbons, or Graham's Mixture (methyl alcohol, benzene, and acetone).

(*b*) Dissolve the grease in the solvent by boiling under a reflux condenser if necessary. Filter hot on a tared glass filtering crucible or other suitable filter medium. A heated filter crucible[3] is frequently helpful. Where the filler is present in large quantities, or otherwise causes excessive difficulty in filtration, it may be necessary to conduct the solution and washing operations in a centrifuge tube, substituting centrifuging for filtration. Wash the residue with hot solvent, adding the washings to the filtrate. Save the filtrate, if a large sample was used for filler determination, for the determination of fatty acids (Section 9 (*a*) and (*b*)).

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[2] 1945 Supplement to Book of A.S.T.M. Standards, Part III, p. 151.

[3] S. H. Hulse and H. L. Thwaites, "Determining the Sediment Content of Fuel Oil," *Industrial and Engineering Chemistry*, Anal. Ed., Vol. 10, p. 678 (1938).

(*c*) Dry the residue on the filter at a suitable temperature, considering the boiling point of the solvent, and weigh.

SOAP

Analysis for Soap Bases

8. (*a*) *Alkali Metal Soap* (*Qualitative*).—Boil a sample of the original grease with water, and filter. Shake the aqueous solution and note whether frothing occurs. Test a portion of this solution with barium chloride and observe for the appearance of curdy precipitate. Both will be evident if alkali metal soap is present. Alkali sulfonates will behave generally as soaps but will be distinguished by the emulsions they cause in the analysis. If sulfonates comprise the soap constituent, it will be necessary to wash the hydrocarbon or ether solutions of them encountered in the analytical separations with sodium chloride solution instead of merely with water.

(*b*) *Soaps of Metals Other than Alkalis* (*Qualitative*).—Dry a portion of the original grease sample in an oven. Digest with boiling benzene, filter, and test the hot filtrate by addition of appropriate reagents; for example, to test for calcium add hot alcoholic oxalic acid solution.

(*c*) *Soap Bases* (*Quantitative*).—Before allocating the fatty acids from soap in greases which contain soaps of two metals, determine quantitatively the most accurately determinable organically combined metal in the soluble portion of the grease. The metal can be determined by conventional inorganic procedures applied to the aqueous solution (*A*) obtained in accordance with Section 9 (*a*). It has been found that lithium in lithium-sodium soap greases can best be determined by the special method described in Appendix II.

Fatty Acids, Free and from Soap

9. (*a*) *Separation of Acids*.—Evaporate the filtrate saved from Section 7 (*b*) to small volume (approximately 25 ml.) in a 250-ml. Erlenmeyer flask and add 20 ml. of benzene (NOTE 1). Some solvents interfere with the chemistry of subsequent separations or cause bad emulsions and these should be completely eliminated by evaporation.

Add 50 ml. of HCl (10 per cent) and several boiling stones to prevent bumping. Digest by boiling under a reflux condenser until the sample is completely disintegrated, 3 hr. is generally sufficient. Formation of a clear oil layer is evidence of such decomposition. Greases difficult to decompose may be resolved by more vigorous boiling, use of additional benzene, or use of a stronger HCl solution. Remove from the condenser and evaporate the benzene in a safe location, avoiding excessive heating so as not to lose volatile oils. Transfer the contents of the cool flask to a separatory funnel, using grease analysis naphtha and distilled water as washing liquids. If asphaltic and tarry matter is present, it will be difficult to complete the transfer with grease analysis naphtha alone. In such case, add small but sufficient amounts of benzene to effect complete transfer.

Allow the solution in the separatory funnel to separate and draw the aqueous layer (*A*),[4] which contains the soap metals as chlorides and glycerin, into another separatory funnel. Wash the naphtha layer (*B*) with 25-ml. portions (NOTE 2) of water containing methyl orange indicator until removal of the mineral acid is complete (NOTE 3). Add these washings to aqueous layer (*A*).

Wash aqueous solution (*A*) with two successive 20-ml. portions of grease analysis naphtha. Combine these two 20-ml. portions of solvent and wash once with 15 ml. of distilled water. Draw off and discard the water. Add this naphtha layer to naphtha solution (*B*). Set aqueous solution (*A*) aside for further examination as described in Section 13.

Add sufficient 0.5 *N* alcoholic KOH to naphtha solution (*B*) (NOTE 4) to keep the alcoholic layer distinctly alkaline to phenolphthalein after vigorous shaking. The conservative addition of water at this point may aid in securing rapid and sharp separation.

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[4] Solutions designated by capital letters in parentheses throughout this method refer to the solutions shown in Fig. 1.

Allow the two solutions to separate sharply and withdraw the lower (alcoholic) layer (*C*) into another separatory funnel. Wash the upper (naphtha) layer (*D*) three times with 30-, 25-, and 20-ml. portions, respectively, of neutral 50 per cent alcohol or until alkali-free. Add these washings to (*C*).

Wash solution (*C*) with 25 ml. of grease analysis naphtha, adding the washings to naphtha solution (*D*). Save solution (*D*) for Section 10 (*a*). Place the alcoholic solution (*C*) in an Erlenmeyer flask or beaker and evaporate to a small volume or substantial dryness to remove the alcohol. Wash the residue of potassium soap into a separatory funnel with hot water. Acidify strongly with HCl, and after the solution has cooled, shake successively with 50- and 25-ml. portions of ethyl ether. If the acids are difficultly soluble, it may be necessary to use additional washings of 25-ml. portions of ethyl ether.

Place the ether solution (*E*) in a separatory funnel and wash twice (or until not acid to methyl orange (NOTE 2)) with 20-ml. portions of water. Discard the water washings. Transfer the ether solution (*E*) (NOTE 5) into a weighed Erlenmeyer flask. Evaporate to dryness on the steam bath, sweeping the surface with air (NOTE 6) to remove the last traces of ethyl ether. Heat the residue for a short time on the steam bath, adding and evaporating 5-ml. portions of acetone to remove the last traces of water. Weigh the dry residue as fatty acids from soap and free fatty acids. Acidic asphaltic or tarry matter, which may have been present in the grease, will be included. In subsequent calculation of soap, correction should be made for free fatty acid as determined in Section 15 (*a*).

NOTE 1.—If fillers are absent or if a separate sample (2 to 3 g.) has been used for the determination of fillers, the separation of acids should be made on a new portion of the original sample.

NOTE 2.—Three washings should be sufficient. A greater number may occasionally be necessary but should be limited as much as possible due to the possibility of small losses of fatty acids through solubility in water.

NOTE 3.—When asphaltic and tarry matter is present in a grease, it may precipitate from the grease analysis naphtha solvent and stick to the inside of the separatory funnel or form a middle layer. In such cases, use small additional rinsings of warm benzene and thus carry this matter along in solution in the analysis.

NOTE 4.—If naphtha solution (*B*) is not too dark, an approximation of the sum of free fatty acids and fatty acids from soap may be obtained for most greases by titrating this solution in the separatory funnel with 0.5 *N* alcoholic KOH, using phenolphthalein as indicator, and using 200 as the assumed neutralization number of the fatty acid.

NOTE 5.—If large amounts of filler were present and a separate sample was used for this determination, it may be necessary to filter this ether solution before transferring it to the Erlenmeyer flask in order to eliminate any filler that may have been carried along during the analysis.

NOTE 6.—If there is reason to believe that the fatty acids are susceptible to oxidation, an inert gas such as nitrogen should be used to sweep out the flask. However, such fatty acids are very infrequently encountered.

(*b*) *Examination of Acids.*—Determine the neutralization number of the fatty acids by adding to a maximum of 1 g. of acids, 50 ml. of neutralized 95 per cent alcohol and titrating with 0.1 *N* KOH, using 1 ml. of phenolphthalein indicator solution, to a sharp pink end point while hot. The neutralization number will be needed in computing soap. Large difference between neutralization and saponification numbers is a clue to the presence of lactones or anhydrides. Very low neutralization numbers may also indicate poor elimination of unsaponifiables. The acids may be used for further chemical or physical examination for identification.

FAT, PETROLEUM AND UNSAPONIFIABLE MATTER

Procedure

10. (*a*) Wash naphtha solution (*D*) from Section 9 (*a*), containing free fat, petroleum, and unsaponifiable matter, with HCl (10 per cent) to decompose small amounts of soap that may still be dissolved in it, and then with water until the mineral acid is removed. If large amounts of filler are present in the grease and a separate sample has been used for the soap separation, it may be necessary to filter this naphtha solution in order to eliminate filler which may have been carried along during the analysis.

The filler so separated should be washed with hot benzene to remove asphaltic and tarry matter and the washings added to the main naphtha solution. Pour the naphtha solution (which will contain benzene if tarry matter, etc. is present) into a weighed 250-ml. Erlenmeyer flask. Evaporate the naphtha and weigh the residue as petroleum oils, free fat, and unsaponifiable matter, which will contain nonacidic asphaltic and tarry matter that may have been carried along in the analysis.

NOTE 7.—Greases for low-temperature operations may contain kerosine, gas oil, or light pale oil which may largely be lost by evaporation in this procedure. In such cases a dilution test, using $CaCl_2$ brine instead of water, may be helpful in estimating the amount of such volatile oils. Light pale oils and heavy gas oils may be retained by carefully controlled weighing at 5-min. intervals when evaporating the solvent in this procedure, the break in the evaporation loss-time curve indicating the end point. In this manner three low-temperature greases made to contain 83 per cent minimum of low-viscosity mineral oil were found to contain 82 to 84 per cent by the proposed method as against 60 to 67 per cent by the A.S.T.M. Method D 128, Analysis of Grease.[5]

(*b*) Determine the neutralization number of this residue and add the equivalent acids to the fatty acids determined in accordance with Section 9 (*a*).

(*c*) Determine the saponification number of this neutralized residue by digestion with alcoholic KOH. Save this saponified material for Paragraph (*d*). Calculate the percentage of fat, assuming a saponification number of 195.

(*d*) If the calculated fat content is over 0.5 per cent, verify the amount of it by separating and weighing its fatty acids (fatty acids × 1.045 = fat) from the saponified mass, by the general procedure outlined in Section 9 (*a*). Save the fatty acids for Section 11 (*c*). For best results the actual separation of fatty acids should be employed even for low fat content.

(*e*) Wash the naphtha solution from Paragraph (*d*) with HCl (10 per cent) to decompose traces of soap, and then with water to remove mineral acid. Evaporate the naphtha solution, dry the residue by addition of 10 ml. of pure isopropyl alcohol and re-evaporation, and weigh the dry residue as petroleum and unsaponifiable matter. Save the residue for Section 11 (*b*).

ASPHALTIC AND TARRY MATTER

Procedure

11. These separations need not be made unless asphaltic and tarry matter was evident during the preceding operations.

(*a*) *Acidic Matter.*—Weigh as large a sample as available of the fatty acids separated in Section 9 (*a*) into a 250-ml. Erlenmeyer flask. Add grease analysis naphtha so the ratio of weight of sample to volume of solvent will be 1:10. Swirl the flask, warming to effect maximum solution, bring to room temperature, and let stand 1 hr. Filter through paper, washing the residue in the flask and on the paper with small amounts of naphtha. Dissolve the material left on the paper and in the flask with warm benzene. Transfer the solution to a weighed flask and evaporate. Cool the residue and weigh as acidic asphaltic and tarry matter.

(*b*) *Unsaponifiable Matter.*—On the petroleum and unsaponifiable matter from Section 10 (*e*) determine the insoluble matter in naphtha as described in Paragraph (*a*) and record as unsaponifiable asphaltic and tarry matter. Save the naphtha solution for Section 12 (*a*).

(*c*) *Saponifiable Nonacidic Matter.*—On the fatty acids from fat separated in Section 10 (*d*), determine the naphtha-insoluble portion as outlined in Paragraph (*a*) and record as saponifiable nonacidic asphaltic and tarry matter.

PETROLEUM AND UNSAPONIFIABLE MATTER (RECOVERY FOR IDENTIFICATION)

Procedure

12. (*a*) Evaporate the naphtha solution from Section 11 (*b*). The solute is petroleum and other unsaponifiable oil, and can be used for its further identification.

(*b*) If the grease contains rosin oil, beeswax, wool grease, spermaceti, montan wax, or other fatty materials containing large percentages of unsaponifiable substances, the oil isolated will contain that unsaponifiable matter and the properties found will differ from those of the petroleum products used in making the grease. Special methods can frequently be devised to suit the individual conditions.

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[5] 1944 Book of A.S.T.M. Standards, Par III, p. 182.

GLYCERIN

Procedure for Qualitative Determination

13. Neutralize a portion of solution (*A*) from Section 9 (*a*) with dry sodium carbonate and add sufficient excess to precipitate metals. Carefully evaporate the mass to to dryness and extract the residue several times with strong alcohol. Filter the combined alcoholic extracts and evaporate the alcohol. The residue will contain most of the glycerin with a little sodium chloride. Confirm the presence or absence of glycerin in this residue by conversion to acrolein.[6]

Procedure for Quantitative Determination

14. (*a*) *Scope.*—This method is intended for the determination of free glycerin in grease, and for combined glycerin present as fat in grease. The method has been tried on greases containing 0.03 to 1.6 per cent glycerin—a range commonly present in greases—and found applicable. The method is reported to be accurate for glycerin, even in presence of ethylene and propylene glycols.

(*b*) *Outline of Method.*—To determine free glycerin in grease, the sample is refluxed with dilute H_2SO_4 and benzene, the benzene evaporated to reduce subsequent emulsion difficulties, the mixture extracted with water, the washings neutralized and then oxidized with potassium periodate,[7] and the resulting solution titrated with standard NaOH solution. Glycerin oxidizes to two moles of formaldehyde and one of formic acid:

$$C_3H_8O_3 + 2KIO_4 \rightarrow 2HCHO + HCOOH + 2KIO_3 + H_2O$$

To determine free glycerin and glycerin combined as fat, NaOH, instead of H_2SO_4, is added before refluxing. After saponifying, the soaps are decomposed by refluxing with mineral acid, the benzene is evaporated, and the glycerin extracted with water. This aqueous solution is then oxidized as described above.

(*c*) *Special Reagents:*

(*1*) *Potassium Periodate, c.p.*

(*2*) *Standard Sodium Hydroxide Solution* (*0.05 N*).

(*3*) *Methyl Red Indicator Solution.*[8]

(*d*) *Procedure for Free Glycerin.*—Weigh 10 g. of sample into a 250-ml. Erlenmeyer flask. Add dilute H_2SO_4 (3 ml. of concentrated H_2SO_4 plus 20 ml. of distilled water) and 20 ml. of c.p. benzene. Reflux on a steam plate until the grease is decomposed. Heat to evaporate the benzene, cool to room temperature, then add 50 ml. of grease analysis naphtha. Transfer the contents of the flask quantitatively to a 500-ml. separatory funnel, washing the flask with 50 ml. of naphtha and 25 ml. of distilled water. Add these washings to the separatory funnel. Shake the contents in the separatory funnel, then allow the two layers to separate. Withdraw the water layer into a second separatory funnel containing 100 ml. of naphtha. Wash the first separatory funnel with two 30-ml. portions of distilled water and add the washings to the second separatory funnel. Shake the second separatory funnel, then allow the contents to separate. Filter the water layer through filter paper into a 300-ml., ground-glass stoppered Erlenmeyer flask. Discard the naphtha solutions. Wash the second separatory funnel once with 20 ml. of distilled water and filter this water into the same Erlenmeyer flask.

To the aqueous solution add 3 drops of methyl red indicator and make almost neutral with 6 *N* aqueous NaOH. Add 2 more drops of the indicator and carefully bring to a copper-colored end point with 0.05 *N* NaOH.

To a clean, 300-ml., ground-glass stoppered Erlenmeyer flask add 2 ml. of concentrated H_2SO_4 dissolved in 100 ml. of distilled water. Make the solution neutral to methyl red as directed above, and use this as a blank on the reagent.

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[6] E. H. Huntress and S. P. Mulliken, "Identification of Pure Organic Compounds," Order I, p. 479 (1941).

[7] G. Hoepe and W. D. Treadwell, *Chemical Abstracts*, Vol. 36, 4058 (1942).

[8] The indicator is red in a neutral or acid solution. An alkaline (aqueous unadulterated) solution is yellow. In the glycerin determination, the solution is copper-colored, corresponding to faint alkalinity.

To each of the flasks add 3 g. of c.p. potassium periodate, tie the stoppers carefully in place, and shake in a mechanical shaker for 2 hr. Remove the excess of potassium periodate by filtering the solution through filter paper (Whatman No. 42 or equivalent) and collect the filtrate in clean, 300-ml., ground-glass stoppered Erlenmeyer flasks. Add 6 drops of methyl red indicator, titrate to a copper-colored end point with 0.05 N NaOH, and record the amount of NaOH used.

(*e*) *Procedure for Free and Combined Glycerin.*—See Paragraph (*b*).

(*f*) *Calculation.*—Calculate glycerin as follows:

$$G = \frac{(M - M_1) \times N \times 0.0921}{S \times 0.92} \times 100$$

where:

G = percentage of glycerin,
M = milliliters of 0.05 N NaOH used to titrate the sample,
M_1 = milliliters of 0.05 N NaOH used to titrate the blank,
N = normality of the NaOH,
S = weight of the sample, and
0.92 is an empirical factor derived from experimental data (not to be confused with 0.0921 which is the stoichiometric glycerin value).

ACIDITY AND ALKALINITY

Coexisting Free Alkali and Free Acid

15. (*a*) *Free Acid.*—Weigh 2 to 10 g. of sample into a 250-ml. Erlenmeyer flask, smearing the grease over the inside of the flask with a glass rod to facilitate disintegration. Add 100 ml. of a neutral mixture of equal volumes of benzene and 98 per cent isopropyl alcohol, and a few glass beads. Agitate the sample or stir with a glass rod to disintegrate the grease if possible. If necessary, the grease and solvent may be heated under a reflux condenser to completely disintegrate the sample (NOTE 8). Promptly titrate the free acid with standard 0.1 N alcoholic KOH, using phenolphthalein indicator. Save this solution for Paragraph (*b*). Run a blank determination at the same time, using all reagents but without the grease sample. Calculate free acid in terms of oleic acid.

NOTE 8.—It is preferable to effect the disintegration of the grease at room temperature whenever possible, since lower free fatty acids of greater reactivity may be present and higher temperatures may accelerate their interaction with the free alkali simultaneously present. To those greases which are difficult to disintegrate, it is advantageous to add the benzene component alone, reflux the mixture for a few minutes, and then add the isopropyl alcohol and continue in the usual manner.

(*b*) *Free Alkali.*—To the neutralized solution remaining after the determination of free acid (Paragraph (*a*)), or to the original solution if initially alkaline, add 20 ml. of 0.5 N aqueous HCl. Boil the mixture under a reflux condenser for 10 min. Add 50 ml. of neutral 98 per cent isopropyl alcohol and promptly titrate the excess acid with standard 0.5 N alcoholic KOH to phenolphthalein. Run a blank at the same time, using all reagents but without the neutralized solution. Calculate free alkali in terms of the carbonate of the predominating soap base.[9]

Free Acid in the Presence of Iron, Zinc, Aluminum, or Other Weak-Base Soaps

16. If samples of iron, zinc, aluminum, or other weak-base soaps are present, a direct determination of free acid is not possible, since these metallic soaps react with KOH.

CALCULATING AND REPORTING

Neutralization Number of Fatty Acids

17. Calculate the neutralization number of the fatty acids as follows:

$$\text{Neutralization number} = \frac{56.1AN}{X}$$

where:

A = milliliters of KOH required for titration,
N = normality of KOH used for titration, and

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[9] Gus Kaufman, "Test Methods for Determining Free Acid and Free Alkali in Greases," Subcommittee on Free Acid and Free Alkali, N.L.G.I., *The Institute Spokesman*, January, 1944.

X = grams of fatty acids that were titrated.

Molecular Weight of Fatty Acids

18. Calculate the molecular weight of the fatty acids as follows:

$$\text{Molecular weight} = \frac{56{,}100}{\text{neutralization number of fatty acids}}$$

Soap in Single-Base Greases

19. Calculate the percentage of soap in single-base greases as follows:

$$\text{Soap, per cent} = \frac{BC}{DE}$$

where:

B = molecular weight of the soap,

C = percentage of fatty acids from the soap,

D = molecular weight of fatty acids from the soap, and

E = number of moles of fatty acid per mole of soap.

Soap in Mixed-Base Greases

20. (*a*) If the grease is soluble in organic solvents, determine the more accurately determinable metal (of the two soaps present) in the solvent-soluble portion. Calculate the soap as follows:

$$\text{Soap, per cent} = \frac{FB}{G}$$

where:

F = percentage of the metal determined,

B = molecular weight of the soap, and

G = molecular weight of the metal determined.

Then calculate the fatty acids necessary to form this amount of soap. Subtract these acids from the total soap fatty acids and calculate the remaining soap fatty acids to soap of the second metal, as in Section 19.

(*b*) If the grease is not soluble in organic solvents, determine the most accurately determinable metal and correct it for free alkali if it is the predominant metal. Then proceed as in Paragraph (*a*). The presence of fillers may introduce complications in the analysis of such a grease.

Soap in Greases Containing Heavy Metal Soaps

21. (*a*) If the soap metals are lead, aluminum, etc., where free fatty acids are undeterminable, determine the organically combined metal, add this to the sum of free fatty acids plus fatty acids from soap, and consider the sum the soap content.

(*b*) It is good practice to check the soap content by calculating the soap fatty acids (free and from soap) to soap. If the acids are greatly in excess of that necessary for the normal soap of the metal, it is advisable to calculate the metal to normal soap and report the remaining acids as ***excess acids.***

MISCELLANEOUS

22. Where a great deal of information on the nature of the grease constituents is available, as in manufacturing control, good use has often been made of simpler and faster methods for estimating soap content. For this purpose ash content, sulfated ash content, alkalinity of ash, and organically combined calcium have been used with various degrees of success. The last of these four methods, involving precipitation of calcium by addition of alcoholic oxalic acid to an Alundum filtered hot benzene solution of a lime soap grease, is the most reliable. Similarly, excess alkali in soda-soap grease has been determined by refluxing with standard alcoholic stearic acid and back-titrating the excess to phenolphthalein. Excess alkali in lime-soap grease has been determined on its hot benzene-insoluble matter.

APPENDIX II

DETERMINATION OF LITHIUM IN LITHIUM-SODIUM SOAP GREASE

Scope

1. This method is applicable to lithium-sodium soap greases yielding an ash that does not contain appreciable amounts of sulfate or phosphate.

Outline of Method

2. The sample is burned at a low temperature. The resulting ash is dissolved in dilute hydrochloric acid and evaporated to dryness. Lithium chloride is selectively dissolved from the residue with acetone,[1] sodium chloride remains undissolved. The lithium chloride is sulfated and weighed.

Procedure

3. (*a*) Weigh 1 to 2 g. of the sample into a platinum dish. Heat over a burner until the grease ignites and allow the sample to burn until only carbon and ash remain. Ignite in an electric muffle at 540 C. until the carbon is removed. If the ignition is carried out at too high a temperature the platinum dish will be attacked by the fused alkali oxides. Cool the residue and dissolve by adding HCl (1:1). Transfer the solution to a 250-ml. beaker and evaporate to dryness on the steam bath.

(*b*) If a small amount of sulfate is present in the ash, as indicated by a qualitative test made on a separate sample, a *slight* excess of barium chloride should be added before the solution is evaporated to dryness. Neither barium sulfate, nor chloride in small amounts, will interfere with the subsequent determinations.

(*c*) Add 25 ml. of anhydrous acetone and 1 drop of concentrated HCl to the dry salts. Pulverize and agitate the mixture with a glass rod for 10 to 15 min. Allow the insoluble matter to settle, then decant the solution through a dry paper (11-cm. No. 42 Whatman filter paper or equivalent) collecting the filtrate in a weighed platinum dish.

(*d*) Add 10 ml. of water and 5 ml. of HCl to the original beaker and again evaporate to dryness on the steam bath. Add 25 ml. of anhydrous acetone and 1 drop of concentrated HCl to the dry salts, repeating the extraction as previously done and adding the filtrate to the main filtrate already in the platinum dish. Dissolve with hot water any salts accidentally transferred to the filter paper and collect these washings in the original beaker. Evaporate this solution to dryness with HCl as above and perform one more acetone extraction on the dry salts, decanting through a clean dry paper, and add this filtrate to the platinum dish. Evaporate the acetone extracts to dryness and weigh. (Three acetone extractions performed in this manner separated 0.095 g. of lithium chloride from a comparable amount of sodium chloride. If a larger amount of lithium salt is expected, the extractions should be repeated until the platinum dish no longer gains in weight.)

(*e*) Moisten the residue with concentrated H_2SO_4 and fume to dryness, removing organic matter by the cautious dropwise addition of concentrated HNO_3. Ignite at about 800 C. for several minutes to remove last traces of SO_3. Cool in a desiccator and weigh as lithium sulfate (Li_2SO_4).

Calculation

4. Calculate the percentage of lithium as follows:

$$\text{Lithium, per cent} = \frac{Li_2SO_4 \times 0.1263}{\text{weight of sample}} \times 100$$

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[1] M. H. Brown and J. H. Reedy, "Determination of Lithium," *Industrial and Engineering Chemistry*, Anal. Ed., Vol. 2, p. 304 (1930).

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON pH MEASUREMENT

The Symposium on pH Measurement was presented at the Fourteenth and Fifteenth Sessions of the Forty-Ninth Annual Meeting of the American Society for Testing Materials in Buffalo, N. Y., on Thursday, June 27, 1946. The Symposium was sponsored by Technical Committee XIII on Hydrogen Ion Determinations of Committee E-1 on Methods of Testing, through a special committee consisting of E. B. Ashcraft, Westinghouse Electric Corp., Chairman, G. A. Perley, D. S. McKinney, G. G. Manov, and F. D. Tuemmler.

Mr. Ashcraft was chairman of the fourteenth session, and Mr. McKinney of the fifteenth session.

The principal purpose of the Symposium was to present the latest theory and practice in the various methods, such as colorimetric and potentiometric, for making pH and closely related measurements. The authors are experts in the field of pH determinations, and their contributions should be very valuable to those who are interested in such work.

The Symposium consisted of the following seven papers:

Recent Advances in the Theory and Use of Glass Electrodes for pH Determinations—Malcolm Dole
Definition of pH and Extension of the Acidity Scale to Nonaqueous Systems—D. S. McKinney, Paul Fugassi, and J. C. Warner
Acid-Base Equilibria in Aqueous and Nonaqueous Solutions—Martin Kilpatrick
The Potentiometric Estimation of Hydrogen Ion Concentration in Nonaqueous Media—L. Lykken
Standard Buffer Solutions—George G. Manov
Precautions to be Observed in Making Colorimetric pH Determinations—F. R. McCrumb
Historical Review of the Hydrogen Electrode and the Calomel Cell in the Measurement of Hydrogen Ions—C. N. Meyers

These papers together with discussion are issued as *Technical Publication No. 73*, entitled "Symposium on pH Measurement."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON OIL PROCUREMENT PRACTICES

The Symposium on Oil Procurement Practices was presented at the Sixth and Eleventh Sessions of the Forty-Ninth Annual Meeting of the Society in Buffalo, N. Y., June 25 and 26, 1946. The Symposium was sponsored by Technical Committee B on Lubricating Oils of A.S.T.M. Committee D-2 on Petroleum Products and Lubricants as a first step in a program laid down by a special study committee of Committee D-2 to explore the possibility of preparing purchase specifications for lubricants.

The principal purpose of this Symposium is to make available to these interested, information about practices in the purchase of lubricants which are now being used with success.

The Symposium consisted of the following seven papers:

Introduction to Symposium on Oil Procurement Practices—T. A. Boyd
Purchasing of Petroleum Oils by the General Electric Co.—Christian Dantsizen
Eastman Kodak Purchase Specifications for Lubricants—C. L. Pope
Airline Oil Procurement Practices—J. T. Hendren
A Recommended Practice for the Purchase of Lubricants—B. C. Voshell
U. S. Navy Oil Procurement Practices—J. P. Costello
Oil Procurement Practice in an Automobile Plant—J. L. McCloud

These papers together with discussion are issued as a separate publication entitled "Symposium on Oil Procurement Practices."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON SPECTROSCOPIC LIGHT SOURCES

The Symposium on Spectroscopic Light Sources was presented at the Third and Fifth Sessions of the Forty-Ninth Annual Meeting of the Society in Buffalo, N. Y., on Tuesday, June 25, at a morning and an afternoon session. Committee E-2 on Spectrographic Analysis sponsored the two sessions, through a committee consisting of E. B. Ashcraft, Westinghouse Electric Corp., Chairman, G. H. Dieke, Johns Hopkins University, and Jacob Sherman, Philadelphia Navy Yard.

Mr. Ashcraft served as chairman of the third session, and H. V. Churchill, Aluminum Company of America was chairman at the fourth session.

This Symposium was organized with the thought that the time had arrived when the field of spectrochemical analysis could best be advanced by a detailed examination of its various subdivisions. The techniques and instruments used in spectrochemical analysis divide themselves more or less naturally into three groups having to do, respectively, with the excitation of the sample, the resolution of the spectrum, and the determination of the relative intensities of the light emitted at various wavelengths. It was thought by the committee that the first of these groups is perhaps the least advanced and that a detailed study in the form of a symposium might be a particularly desirable contribution at this time.

The Symposium consisted of the following four papers:

The Present Status of Excitation in Spectrographic Analysis—B. F. Scribner
A Study of the Controlled Spectrographic Spark Source—J. H. Enns and R. A. Wolfe
Some Properties of Gas Discharges Used as Spectral Sources—R. C. Mason
Short Period Behavior of Spectroscopic Light Sources—G. H. Dieke

These papers, together with invited discussions prepared by several well-known practicing spectroscopists, are issued as ***Technical Publication No. 74***, entitled "Symposium on Spectroscopic Light Sources."

SUMMARY OF PROCEEDINGS OF THE PITTSBURGH SPRING MEETING

SYMPOSIUM ON ATMOSPHERIC EXPOSURE TESTS ON NON-FERROUS METALS

DISCUSSION ON STATISTICAL QUALITY CONTROL IN ITS APPLICATION TO SPECIFICATION REQUIREMENTS

The 1946 Spring Meeting of the Society was held in Pittsburgh, Pa., at the William Penn Hotel, on Tuesday, February 26 and Wednesday, February 27, 1946 in conjunction with the Spring Group Meetings of A.S.T.M. committees. The Spring Meeting consisted of two technical symposiums, one on atmospheric exposure tests on non-ferrous metals and the other on statistical quality control.

Symposium on Atmospheric Exposure Tests on Non-Ferrous Metals:

Two sessions were devoted to the Symposium on Atmospheric Exposure Tests on Non-Ferrous Metals—afternoon and evening of Wednesday, February 27. The symposium was sponsored by A.S.T.M. Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys, and was arranged by W. H. Finkeldey of Singmaster & Breyer, chairman of Subcommittee VI on Atmospheric Corrosion. J. J. Bowman, Aluminum Company of America, cooperated for the Pittsburgh District Committee.

The purpose of the Symposium is to present the data resulting from the extensive atmospheric corrosion tests carried out by Committee B-3 over a period of ten years, and to evaluate these data so that American industry may make practical use of the results. All authors are leading authorities in their own particular fields, thus making a valuable contribution to industry. The Symposium consisted of the following papers:

Introduction—W. H. Finkeldey
The Corrosion of Rolled Zinc in the Outdoor Atmosphere—E. A. Anderson
The Behavior of Nickel and Monel in Outdoor Atmospheres—W. A. Wesley
Resistance of Copper Alloys to Atmospheric Corrosion—A. W. Tracy
The Use of Lead and Tin Outdoors—George O. Hiers
The Resistance of Aluminum-Base Alloys to Atmospheric Exposure—E. H. Dix, Jr. and R. B. Mears
Tracking Troubles in Atmospheric Corrosion Testing—P. S. Olmstead, W. E. Campbell, and H. G. Romig

Mr. Finkeldey was chairman of the afternoon session and Mr. Bowman chairman of the evening session.

Discussion on Statistical Quality Control in Its Application to Specification Requirements:

The Discussion on Statistical Quality Control in Its Application to Specification Requirements was held on Tuesday evening, February 26, 1946. Arrangements for the Discussion were made by the Pittsburgh District Committee, headed by Thomas Spooner, Chairman, and P. G. McVetty, Secretary, both of Westinghouse Electric Corp., assisted by F. T. Mavis, Carnegie Institute of Technology. H. F. Dodge, Bell Telephone Laboratories, Inc., acted as chairman of the session.

The widespread application of statistical quality control methods during the war years testifies to the increasing importance of quality control in production. The authors of the papers are outstanding authorities in this field, and their contributions should prove to be of great value to industry. The papers included are as follows:

Introduction—H. F. Dodge
Use of Statistics in Writing Specifications—Casper Goffman and Joseph Manuele
Dollars for your Thoughts—Leslie E. Simon

The papers in both the above symposiums elicited discussion which contributed greatly to the information contained in the papers. The papers complete with discussion have been issued by the A.S.T.M. as separate publications entitled, respectively, "Symposium on Atmospheric Exposure Tests on Non-Ferrous Metals," and "Discussion on Statistical Quality Control in Its Application to Specification Requirements."

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